



# CEREAL CHEMISTRY

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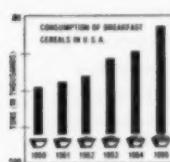
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New Edition

## AMERICA LIKES BREAKFAST FOODS

Let no one doubt the popularity of breakfast cereals among Americans. The chart below traces the consumption of these fine foods between 1930 and 1955. During that period annual consumption rose by 76,000 tons. In just one year, 1955, Americans ate 2½ lbs. of hot and 4.8 lbs. of cold cereals per person!



Why are breakfast cereals so well-liked? They are tasty; they are easily served; they appeal to busy homemakers, as well as institutional dietitians, because they are readily available in a variety of flavors at a modest cost. They add interest and value to an important but sometimes neglected meal—breakfast. Their use is extending to between-meal and party snacks, too.

Many grains are processed to make breakfast cereals: wheat, corn, oats, rice. Eaten with fruit and milk or light cream, they contribute an excellent combination of basic, flavorful, nutritious foods to the diet.

Better Foods for Better Health  
Through Restoration

The science of nutrition has advanced rapidly. In the manufacturing process of some cereals, some of the essential "B" vitamins and minerals are subject to some loss, just as with other foods.

These losses are inescapable when such grains are prepared for human use. When this became known, manufacturers acted to overcome the losses. They adopted restoration.



Restoration simply means that certain important vitamins and minerals are restored to the cereal food during processing, so that the vitamin and mineral values in the finished product are generally equal to the whole grain values of those elements. Wheat, corn and rice products are customarily so treated. Vitamins B<sub>1</sub> (thiamine), B<sub>2</sub> (riboflavin), niacin (another "B" vitamin), and the mineral, iron, are those most widely restored. Vitamins C and D are also sometimes added.

Pre-sweetened cold cereals emphasize the nutritional importance of added vitamins. Increased calories require more "B" vitamins for best utilization of the food.

## Why the Vitamins are Important

Physicians and diet experts have proved that vitamins are essential to prevent certain deficiency diseases and to contribute to robust good health.

**Vitamin B<sub>1</sub> (thiamine)** helps build and maintain physical and mental health. It is essential for normal appetite, intestinal activity, and sound nerves. A lack of this vitamin leads to beriberi, a rarity in the U. S. A., but still a very serious health problem in other parts of the world.

**Vitamin B<sub>2</sub> (riboflavin)** is essential for growth. It helps to keep body tissues healthy and to maintain proper function of the eyes.

**Niacin** is needed for healthy body tissues. Its use in the American diet has been largely responsible for the virtual disappearance of pellagra, a serious disease.

**Vitamin D** helps children develop normal teeth and bones. It prevents the development of certain abnormal bone conditions in adults.

**Iron** is essential for making good red blood and for the prevention of nutritional anemia.



## Where Do the Vitamins Come From?

At about the same time that processing losses in breakfast cereals became known, other developments in the scientific world made available ample supplies of vitamins at economical prices. Thus, the nutritional contribution of some breakfast cereals could be, and was, greatly improved through restoration.

Since the early days of breakfast food restoration and of white flour and white bread enrichment, the world-famous firm of Hoffmann La Roche has supplied top quality vitamins by the tons. Pioneering work in its laboratories and by its collaborators resulted in the "duplication" of some of nature's extremely complex substances. First, the chemical composition of the vitamin was learned. Second, the pure substance was isolated. Third, the "duplicate" was made by synthesis. And fourth, the laboratory techniques were extended to large scale commercial operations.

The manufactured "duplicate" is identical chemically and in biological activity with nature's own product. A vitamin is still a vitamin regardless of whether nature or man made it. So efficient is large-scale manufacturing, that vitamins are sold at a lower cost than if they were extracted from natural sources.



This article is one of a series devoted to the story of vitamin-enriched or restored cereal products: white flour, white bread and rolls, corn meal and grits, macaroni products, white rice, breakfast cereals, farina. Reprints of this article, of any other in the series, or of all are available without charge. Please send your request to the Vitamin Division, Hoffmann-La Roche Inc., Nutley 10, New Jersey. In Canada, Hoffmann-La Roche Ltd., 1956 Bourdon Street, St. Laurent, P. Q.

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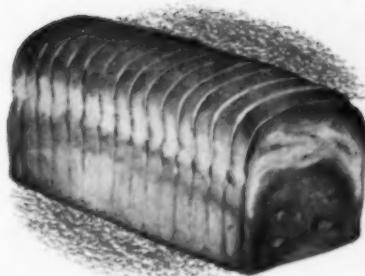
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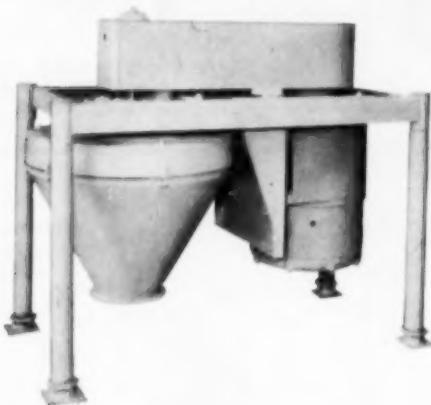
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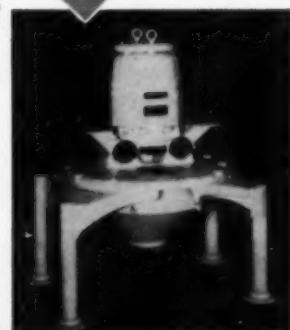


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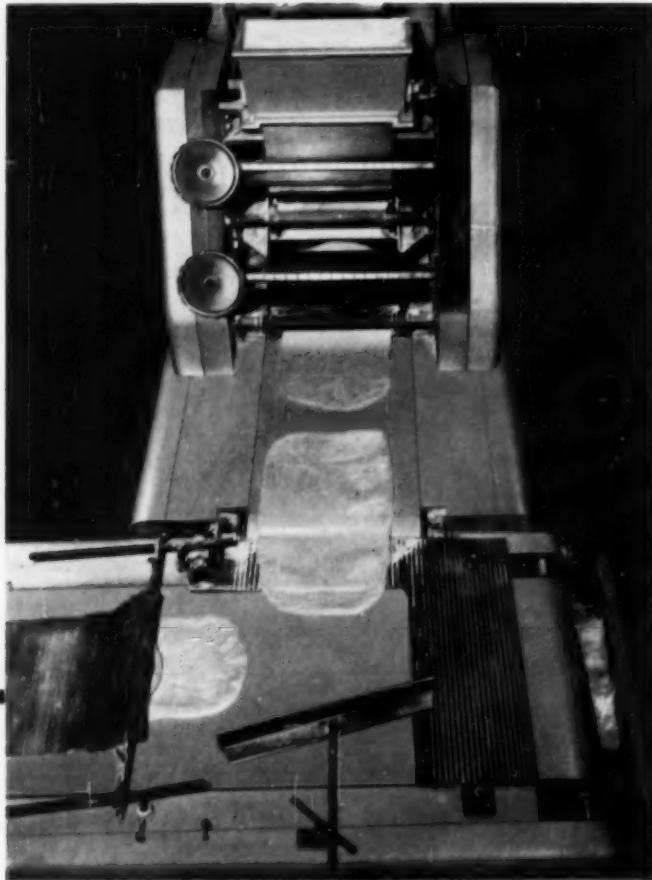
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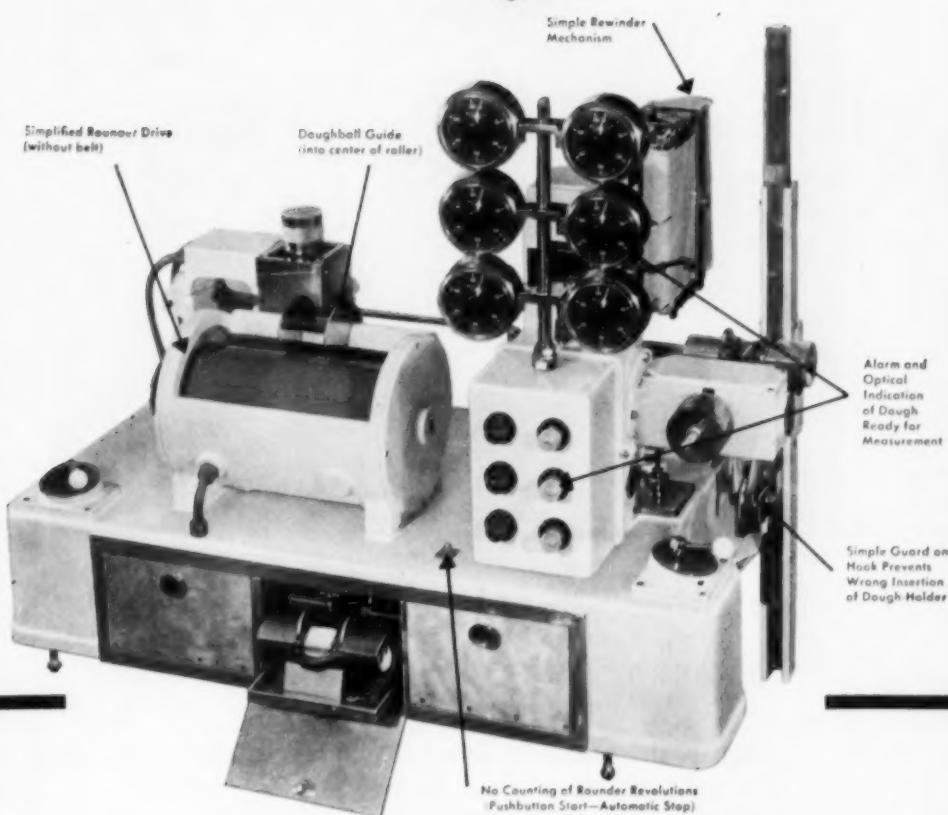
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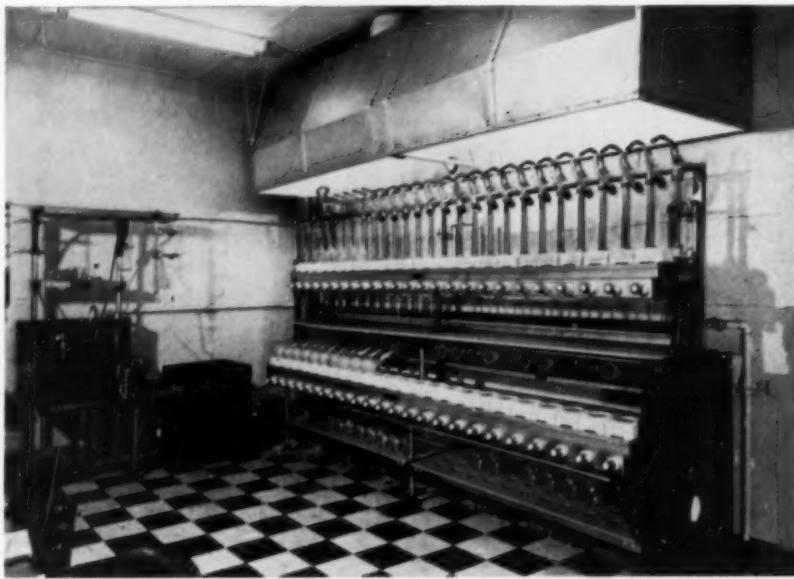
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# CEREAL CHEMISTRY

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NO. 6

## A THEORETICAL STUDY OF THE MECHANISM OF MOISTURE DIFFUSION IN WHEAT<sup>1</sup>

H. A. BECKER AND H. R. SALLANS

### ABSTRACT

The properties of the coefficient for the diffusion of moisture out of wheat are directly related to the moisture desorption isotherm. A three-dimensional flow mechanism applies at moistures above the critical level (10 to 12% dry basis), while at moistures below this critical level the properties of the diffusion coefficient are best explained by a two-dimensional mechanism. The three-dimensional mechanism is based on the equation for viscous flow through a porous medium, and the desorption equilibrium water vapor pressure gradient is used as the driving potential. The two-dimensional mechanism is based on the equation for flow of an adsorbed film, and employs the two-dimensional film pressure as the driving potential. The evidence presented indicates that the adsorbent molecules are the structural units of wheat which are involved in moisture sorption and that they control the rate of diffusion.

The authors have made studies of the moisture desorption isotherm of wheat (5) and of the coefficient for the diffusion of moisture out of wheat (4). In the present paper a number of mechanisms for moisture diffusion in wheat are hypothesized in which the properties of the diffusion coefficient are directly and quantitatively related to the desorption isotherm. A physical interpretation of both the statics and dynamics of moisture desorption in wheat is thereby provided. The theoretical picture so obtained should be generally useful in interpreting the diffusion of moisture through polar organic solids.

### The Driving Potential in Diffusive Flow

Diffusion in solids is a complex process, and although a practical empirical description is sometimes easy, theoretical interpretation of the diffusive flow mechanism is generally difficult. To be properly termed diffusion, any net flow of the diffusing species must be due to a gradient in concentration. In the specific case of diffusion of fluid through a solid, it is evident that such a flow can result only if the interaction between the solid and the diffusing species is sufficiently

<sup>1</sup> Manuscript received March 21, 1957. Contribution from the National Research Council of Canada, Prairie Regional Laboratory, Saskatoon, Saskatchewan. Issued as N. R. C. No. 4528 and No. 342 of the Associate Committee on Grain Research, Canada.

strong to cause a variation in the fugacity of the diffusing molecules with concentration. Consequently, diffusion of a fluid through a solid may be regarded as a movement of adsorbed phase through a solid, adsorbing medium. The mechanism of the diffusive flow of the adsorbed phase may be expected to depend on the state of the adsorbed molecules and on the physical and chemical structure of the solid. Two distinct types of flow mechanism can be hypothesized: (i) if the adsorbate completely fills the adsorbent micropores the flow should be three-dimensional and mathematically similar to flow of fluid through a porous medium, the pressure gradient causing flow resulting from a gradient in concentration; or (ii) under other circumstances the adsorbate may be better pictured as a film coating the internal adsorbent surfaces, and the flow will then correspond to the equations for two-dimensional transport of adsorbed molecules over a solid surface. It is probable that the type of flow occurring depends on the concentration of the adsorbate and the nature of the adsorbent.

If the flow is three-dimensional, the equations for viscous flow of fluid through a porous medium should apply, and the adsorbate vapor-pressure is the driving potential. This pressure is given as a function of concentration by the desorption isotherm. The authors (5) have determined the desorption isotherm of wheat at temperatures of 25° and 50°C. The isotherm was found to be sigmoid in shape, of type IV according to the classification of Brunauer, Emmett, and Teller (7), and was described mathematically as follows:

(i) In the initial curved region,  $0.04 < f < 0.2$ , by the two-constant Brunauer, Emmett, Teller (B.E.T.) equation

$$\frac{m}{m_m} = \frac{cf}{[1-f][1+(c-1)f]} \quad (1)$$

where  $f$  = relative water vapor pressure;

$m$  = moisture content, dry basis, in equilibrium with the relative vapor pressure  $f$ , g/g;

$m_m$  = moisture content, dry basis, corresponding to the first adsorbed molecular monolayer, g/g;

$c$  = a constant;

and at 25°C.  $m_m = 0.0780$  and  $c = 22.9$ ;

(ii) in the intermediate linear region,  $0.12 < f < 0.65$ , by the equation

$$m - m_0 = af \quad (2)$$

where  $m_0$  and  $a$  are constants, and at 25°C.  $m_0 = 0.0483$  and  $a = 0.1715$ ;

(iii) in the final curved region,  $0.5 < f < 0.95$ , by the Smith equation (11)

$$\frac{w - w_b}{w'} = \ln \frac{1}{1-f} \quad (3)$$

where  $w$  = moisture content, wet basis, in equilibrium with the relative vapor pressure  $f$ , g/g;

$w_b, w'$  = constants;

and at 25°C.  $w_b = 0.0845$  and  $w' = 0.0514$ .

In a two-dimensional flow of an adsorbed film, the two-dimensional thermodynamical equivalent of the three-dimensional fluid pressure, as pointed out by Babbitt (2), is the film pressure,  $\pi$ .<sup>2</sup> The equation for this type of flow is, according to Babbitt:

$$J = -\eta c \frac{\delta \pi}{\delta x} \quad (4)$$

where  $J$  = mass rate of flow of adsorbate, g/cm.<sup>2</sup> sec.;

$c$  = adsorbate concentration, g/cm.<sup>3</sup>;

$\delta \pi / \delta x$  = film pressure gradient, dynes/cm.<sup>2</sup>; and

$\eta$  = a resistance coefficient.

The film pressure is thermodynamically defined as the decrease in free surface energy of a solid surface caused by the adsorption of a gas or vapor, and can be estimated from the following integral of the Gibbs adsorption equation (1):

$$\pi = \frac{RT}{V\Sigma} \int_{0}^{P} \frac{v}{p} dp \quad (5)$$

where  $\pi$  = film pressure, ergs/cm.<sup>2</sup> or dynes/cm.;

$R$  = gas constant =  $8.315 \times 10^7$  ergs/mol. °K;

$T$  = absolute temperature, °K;

$V$  = molar volume of adsorbed gas, cm.<sup>3</sup>/mol.;

$v$  = volume of gas adsorbed per unit mass of adsorbent, cm.<sup>3</sup>/g.;

$p$  = equilibrium pressure of adsorbed gas, dynes/cm.<sup>2</sup>;

$\Sigma$  = specific surface area of adsorbent, cm.<sup>2</sup>/g.;

For the present purposes this equation is conveniently written

$$\pi = \frac{RT}{M\Sigma} \int_{0}^{f} \frac{m}{f} df \quad (6)$$

where  $M$  is the molecular weight of the adsorbate, g/mol. The integral can be evaluated graphically or analytically, depending on whether isotherm equations for the relation between  $m$  and  $f$  are available. For

<sup>2</sup> Called by Babbitt the "spreading pressure."

the desorption of water from wheat, equilibrium data are lacking for  $f < 0.04$ , and it will therefore be assumed that the B.E.T. equation is valid in the entire range  $0 < f < 0.3$ . On substituting the B.E.T. expression for  $m$  in equation 6 and integrating, there is obtained

$$\pi = m_m \frac{RT}{M\Sigma} \ln \frac{1 + (c - 1)f}{1 - f} \quad (7).$$

Similarly, integration in the region where  $m$  and  $f$  are linearly related gives

$$\pi = \pi_1 + \frac{RT}{M\Sigma} \left[ a(f - f_1) + m_0 \ln \frac{f}{f_1} \right] \quad (8),$$

where  $f_1$  is in the region of linearity between  $m$  and  $f$  and  $\pi_1$  is the corresponding value of  $\pi$ . The value of  $\pi_1$  may be calculated at the lower limit of the linear region from the B.E.T. expression for  $\pi$ , equation 7. The integration at high values of  $f$ , in the final curved region of the desorption isotherm, is best completed graphically as the Smith expression for  $\pi$  is too cumbersome for analytical integration.

There is no direct method for determining the specific surface area  $\Sigma$  in the equations for the film pressure. The area will therefore be estimated from the moisture content  $m_m$  which, according to the B.E.T. theory, corresponds to a molecular monolayer of adsorbate. If it is assumed that the adsorbed water molecules are packed as in liquid water, the area  $\sigma_m$  occupied by a molecule in the first monolayer is

$$\sigma_m = \left( \frac{M}{\rho N} \right)^{2/3} \quad (9),$$

where  $\rho$  = density of liquid water, g/cm.<sup>3</sup>;

$N$  = Avogadro's number =  $6.023 \times 10^{23}$  molecules/mol.

Hence

$$\Sigma = \sigma_m N \frac{m_m}{M} \quad (10).$$

At 25°C,  $m_m = 0.078$  g/g, and  $\sigma_m = 9.65 \text{ \AA}^2$ , whence  $\Sigma = 253$  sq. meters per g., dry basis.

The film pressure  $\pi$  at constant temperature is usually determined as a function of the average adsorbent surface area,  $\sigma$ , per adsorbed molecule:

$$\sigma = \sigma_m \frac{m_m}{m} \quad (11),$$

where  $\sigma$  is expressed in  $\text{\AA}^2$  per molecule. The resulting  $\pi - \sigma$  isotherm has the same thermodynamical significance for a two-dimensional film as the P-V isotherm has for a three-dimensional fluid (1, 2). Figure 1 shows the  $\pi - \sigma$  isotherm for water on wheat, as determined

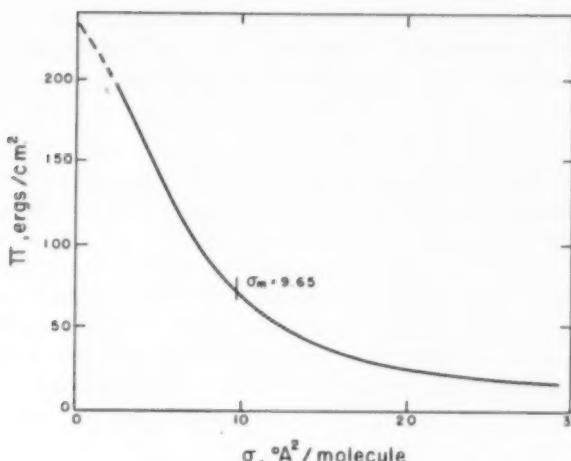


Fig. 1. The  $\pi - \sigma$  isotherm of water on wheat at 25°C.

from the moisture desorption equilibrium data at 25°C. Harkins and Jura (1) in a review have discussed the interpretation of the  $\pi - \sigma$  isotherm in terms of the state of the adsorbed film. The  $\pi\sigma - \pi$  isotherm is frequently informative, and is shown for the present case in Fig. 2.

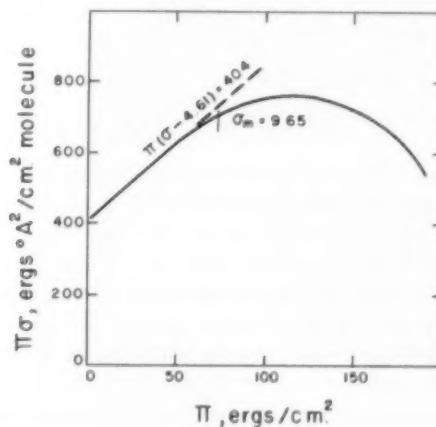


Fig. 2. The  $\pi\sigma - \pi$  isotherm of water on wheat at 25°C.

At low film pressures the relation is linear, and follows the equation of an imperfect two-dimensional gas;

$$\pi(\sigma - \sigma_0) = kT \quad (12).$$

The value of  $\sigma_0$  from Fig. 2 is  $4.61 \text{ \AA}^2$  per molecule. This equation is followed to approximately  $\pi = 60 \text{ ergs/cm.}^2$ . At higher film pressures a long condensed region is exhibited. For condensed phases the equation of state is

$$\pi = a - b\sigma \quad (13),$$

where  $a$  and  $b$  are constants which assume different values for each of the possible condensed phases (1). It is noteworthy that the  $\pi - \sigma$  isotherm of water on wheat is remarkably similar to that of water on anatase ( $TiO_2$ ) at the same temperature (see 1). Harkins and Jura (1) state in their review that in all systems studied to that time (1946), values of  $\pi$  at  $25^\circ\text{C}$ . for water on porous and nonporous polar solids extrapolated to the limit  $f=1$  lay in the range 180 to  $250 \text{ ergs/cm.}^2$ , and Fig. 1 indicates that water on wheat is "normal" in this respect;

$$(\pi)\sigma = 0, 25^\circ\text{C.} = 240 \text{ ergs/cm.}^2$$

### The Three-Dimensional Flow Mechanism

It was stated in the preceding section that if the diffusive flow is a three-dimensional transport of adsorbed molecules through a micro-pore system, the equations for macroscopic flow of fluid through a porous medium should apply, with the difference that the pressure gradient causing flow is due to a gradient in concentration. We shall attempt to describe the three-dimensional flow by the following equation, which was developed from the data of many investigators and is applicable to viscous flow of liquids through randomly packed fixed beds and suspensions of spheres (3):

$$J = \frac{-D_s^2 \rho \epsilon^{4.75}}{18\mu(1-\epsilon)} \cdot \frac{dp}{dx} \quad (14),$$

where  $D_s$  = diameter of spherical particle, cm.;

$\rho$  = liquid density, g/cm.<sup>3</sup>;

$\mu$  = liquid viscosity, g/cm. sec.;

$\epsilon$  = fractional pore space;

$dp/dx$  = pressure gradient, dynes/cm.<sup>2</sup> cm.; and

$J$  = mass liquid flow rate, g/cm.<sup>2</sup> sec.

This equation describes a thoroughly investigated macroscopic system and contains no arbitrary, variable factors. Furthermore, the model pore system adopted by its use, however idealized, should at least approach reality. To apply the equation to the diffusion of moisture through wheat, the following assumptions are made: (i) the structural unit of wheat which effectively controls the flow of moisture can be idealized as a solid, impermeable sphere; and (ii) the micropore system is created entirely by the penetration of water between the structural

units. As a consequence of the second assumption, the fractional pore space is given by

$$\epsilon = \frac{m/\rho}{m/\rho + 1/\rho_s} = \frac{m}{m + 1/\rho_s} \quad (15),$$

where  $\rho_s$  is the density of moisture-free wheat, g/g, and the density,  $\rho$ , of water is taken as unity. It may be noted that the two assumptions partly contradict each other, since solid spheres cannot be packed to give fractional pore spaces smaller than about 0.26, or randomly packed to give fractional pore spaces less than about 0.35. It will be assumed, therefore, that low fractional pore spaces are attained by deformation of the structural units, without, however, changing the value of the constant in equation 14.

The pressure gradient in equation 14 is the vapor pressure gradient, and is related to the moisture gradient by

$$\frac{dp}{dx} = \frac{\delta p}{\delta m} \cdot \frac{dm}{dx} = p_0 \frac{\delta f}{\delta m} \cdot \frac{dm}{dx} \quad (16),$$

where  $p_0$  is the saturation water vapor pressure, and  $\delta f/\delta m$  is obtainable from the desorption isotherm. Hence

$$J = - \frac{D_s^2 \rho p_0 \epsilon^{4.75}}{18\mu (1-\epsilon)} \cdot \frac{\delta f}{\delta m} \cdot \frac{dm}{dx} \quad (17).$$

This gives for the diffusion coefficient

$$D = \frac{D_s^2 \rho p_0 \epsilon^{4.75}}{18\mu (1-\epsilon)} \cdot \frac{\delta f}{\delta m} \quad (18),$$

where the coefficient is defined on the basis of the moisture gradient, as in the author's practical mathematical treatment of the drying of the wheat kernel (4).

Figure 3 shows the factors  $\delta f/\delta m$  and  $\epsilon$  as functions of moisture content for desorption at 25°C., where  $\delta f/\delta m$  was obtained by differentiation of the  $m-f$  isotherm equations 1, 2, and 3. Figure 4 shows the resulting variation of the diffusion coefficient with moisture content, as given by equation 18. The figures suggest that the constancy of the diffusion coefficient in the moisture range 14 to 30%, dry basis, is due chiefly to a balance between the effects of  $\delta f/\delta m$  and the fractional pore space.

The effect of temperature on the group  $p_0\rho/\mu$  in equation 18, where the viscosity  $\mu$  may be assumed to be that of liquid water, corresponds to an energy of activation of 13.8 k.cal/mol. If the variation of  $\delta f/\delta m$  with temperature, as given by the desorption isotherms at 25° and 50°C., is also considered, energies of activation of 11.5 to 13.8 k.cal/mol., depending on the moisture content, are obtained. These energies

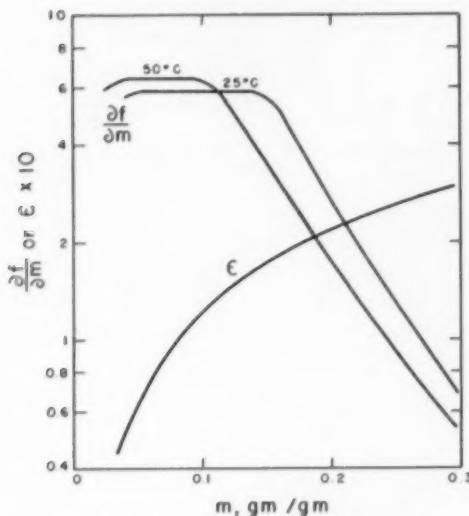


Fig. 3. The factors  $\frac{\partial f}{\partial m}$  and  $\epsilon$  as a function of the moisture content of wheat at  $25^{\circ}\text{C}$ .

of activation are in reasonably good accord with the experimental value, 12.9 k.cal/mol. (4), found for the wheat sample used in determining the desorption isotherms.

#### The Two-Dimensional Flow Mechanism

In the two-dimensional diffusive flow mechanism proposed by Babbitt (2), an expression for the diffusion coefficient is obtained by substituting in equation 4 an appropriate expression for the film

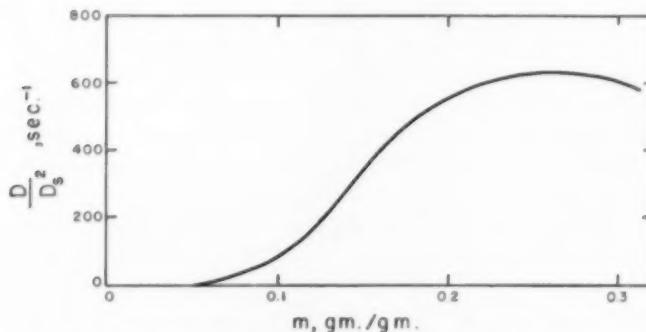


Fig. 4. The diffusion coefficient as a function of moisture content, dry basis, according to the three-dimensional mechanism.

pressure  $\pi$  in terms of moisture content. Equation 4 is written

$$J = -\eta \rho_e m \frac{\delta \pi}{\delta x} \quad (19),$$

where  $\rho_e m$  is equal to the adsorbate concentration. It will be assumed that the density  $\rho_e$  is constant with a value of 1.37 g/cm.<sup>3</sup>, corresponding to 10% moisture content, dry basis. The film pressure,  $\pi$ , is expressed in terms of moisture content by a conventional  $m-f$  isotherm equation or by a  $\pi-\sigma$  equation of state. On differentiating equation 6 and substituting the resulting value of  $d\pi/dx$  in equation 19, we obtain

$$J = -1.37 \eta \frac{RT}{M\Sigma} \frac{m^2}{f} \frac{\delta f}{\delta m} \frac{dm}{dx} \quad (20),$$

whence

$$D = 1.37 \eta \frac{RT}{M\Sigma} \frac{m^2}{f} \frac{\delta f}{\delta m} \quad (21).$$

Expressions for computing the diffusion coefficient as a function of moisture content can be obtained from this equation by evaluating  $f$  and  $\delta f/\delta m$  from the isotherm equations 1, 2, and 3; e.g., the B.E.T. equation gives

$$\frac{D}{\eta} = 1.37 \frac{RT}{M\Sigma} \frac{mm_m}{\sqrt{(m-m_m)^2 + (4/c)mm_m}} \quad (22).$$

On the other hand, the  $\pi-\sigma$  equations of state give  $\pi$  as a function of moisture content by the substitution  $\sigma = \sigma_m m_m/m$ ; e.g., for an imperfect two-dimensional gas equation 12 gives

$$\frac{D}{\eta} = 1.37 \frac{kT}{\sigma_0} \frac{\frac{\sigma_m m_m}{\sigma_0} m}{\left( \frac{\sigma_m m_m}{\sigma_0} - m \right)^2} \quad (23).$$

Figure 5 shows as a function of moisture content the diffusion coefficient at 25°C. computed from the desorption isotherm according to equation 21. The figure indicates that the two-dimensional mechanism describes the experimentally observed behavior of the diffusion coefficient best at moistures below the critical level 10 to 12% dry basis. In agreement with experiment, the diffusion coefficient is practically constant at moistures between 7 and 10% (4). However, no marked increase in the coefficient is predicted in passing through the critical moisture range, and at moistures above 16% the coefficient falls steadily instead of remaining nearly constant.

The theoretically predicted behavior of the diffusion coefficient

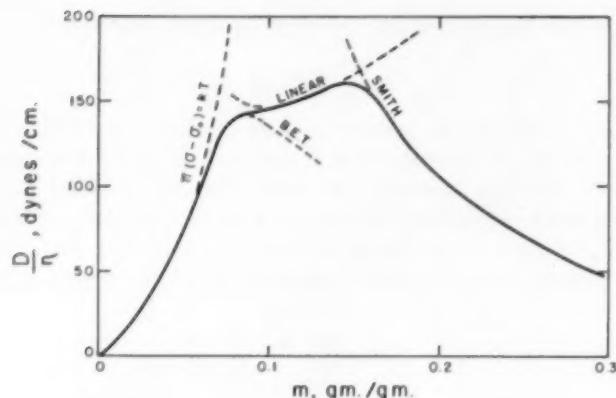


Fig. 5. The diffusion coefficient as a function of moisture content, dry basis, according to the two-dimensional mechanism.

provides a useful basis for elucidating the apparent surface moisture content, as found by the authors in their experimental study of the diffusion coefficient (4). Consider the simple case of stationary state diffusion through a membrane of thickness  $x_0$  subject to the boundary conditions  $m = 0$  at  $x = 0$  and  $m = m_0$  at  $x = x_0$ . For this diffusion

$$J = -D \frac{dm}{dx} \quad (24),$$

whence

$$\frac{x}{x_0} = - \frac{\int_0^m D dm}{J x_0} \quad (25).$$

The integral in this equation may be obtained from Fig. 5, and the equation then determines  $m$  as a function of  $x/x_0$ , as shown in Fig. 6. In the range of moistures 7 to 10% the diffusion coefficient is nearly constant (4), and hence at a given value of  $m_0$ ,  $dm/dx$  is also nearly constant. Extrapolation of the linear relation between  $m$  and  $x/x_0$  to  $x/x_0 = 0$  gives for the apparent surface moisture content  $m_s = 0.047$  g/g, which may be compared with the value 0.052 g/g experimentally determined under nonstationary state conditions (4). Hence, as long as  $m_0$  is greater than about 7%, the stationary state diffusion is given by

$$J = -D \frac{m_0 - m_s}{x_0} \quad (26),$$

where  $D$  is constant. This explanation of the apparent surface moisture

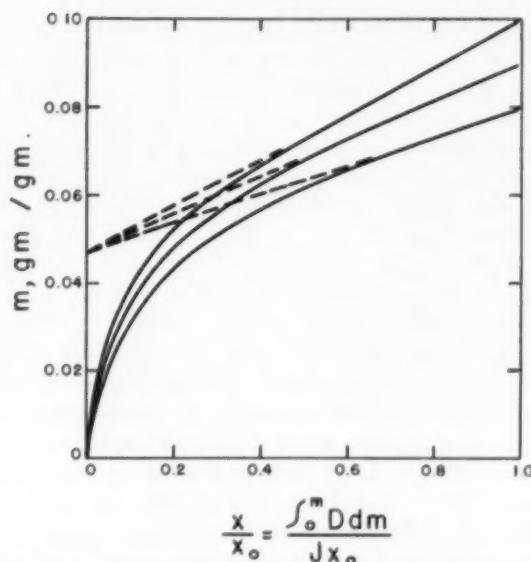


Fig. 6. The distribution of moisture content in steady state diffusion through a film of thickness  $x_0$ , in which the diffusion coefficient is related to moisture content by Fig. 5. Extrapolation of the linear regions of the curves to  $x/x_0 = 0$  gives the apparent surface moisture content.

content applies in any region where the diffusion coefficient is independent of moisture content.

### Discussion

It appears from the evidence which has been considered that the diffusive flow of moisture in wheat is controlled by the three-dimensional mechanism at moistures above the critical level 10 to 12%, dry basis, and by the two-dimensional mechanism at moistures below this critical level. To obtain a clearer physical picture of these mechanisms a knowledge of the size and nature of the structural units of wheat which are involved in moisture desorption is necessary. The hypothesized mechanisms can then be interpreted in terms of the apparent properties of the structural units.

An estimate of the size of the structural units can be obtained from the equation for the three-dimensional mechanism and also from the B.E.T. surface area. At 25°C. the diffusion coefficient in the moisture range 14 to 30%, dry basis, is  $D = 10^{-7}$  cm.<sup>2</sup>/sec. (4), and, from Fig. 4,  $D/D_s^2 = 600$  sec.<sup>-1</sup>. Hence  $D_s = 1.29 \times 10^{-5}$  cm. = 1290 Å. The corresponding surface area is  $\Sigma = 6/\rho_s D_s = 32.8$  sq. meters per g., dry

basis, where  $\rho_s = 1.42 \text{ g/cm}^3$  is the density of moisture-free wheat. On the other hand, the B.E.T. theory gives a surface area of 253 sq. meters per g., which corresponds to a spherical structural unit with a diameter of 164 Å. In view of the assumptions and uncertainties involved in the application of the equation for the three-dimensional mechanism and in calculating the diffusion coefficient, it does not appear that any definite significance can be attached to the nearly eightfold difference in the sizes of the structural unit calculated by the two methods. The B.E.T. method indicates a structural unit in the range of sizes of macromolecules such as proteins, indicating that water sorption in wheat occurs on the "surfaces" of the adsorbent molecules. This is in agreement with the experimentally observed fact that water sorption by cereals and their constituents, such as proteins, starch, and cellulose, is essentially independent of the state of subdivision and appears to be a specific molecular property of the adsorbent itself (6, 8, 9). Hence, it appears that desorption of moisture from wheat is controlled, statically and dynamically, by the adsorbent molecules, since the diffusive flow should be governed by the smallest structural units around which the adsorbate is effectively distributed.

On the basis of these considerations the mechanism of moisture sorption and diffusion in wheat appears to be as follows. At low moistures the water molecules are powerfully bound to specific sorption sites and diffusion consists of site-to-site migration of activated molecules; hence, the two-dimensional mechanism is applicable. However, as the moisture content increases the primary sites become saturated and "multilayer" formation commences. Since the adsorbent is structurally a three-dimensional aggregate of macromolecules, it appears that as multimolecular adsorption proceeds a point is reached where entering water molecules are held in overlapping force fields whose net effect is essentially nondirectional. From this point on the three-dimensional mechanism applies. It is evident that the transition in mechanism should be accompanied by a rather marked change in the diffusion coefficient, as is experimentally shown by the large increase in the coefficient in passing through the critical range of moistures 10 to 12%, dry basis (4). Finally, in further adsorption the three-dimensional structure of adsorbent molecules is forcibly expanded in space by the pressure of the entering adsorbate molecules, while the intermolecular voids are at all times filled by a continuous adsorbate phase.

The following rather speculative calculation illustrates an interesting application of the present results to a material other than wheat.

The authors (4) have determined the coefficient for diffusion of moisture out of wheat in the moisture range 7 to 10%, dry basis, at temperatures of 50° and 81°C., obtaining values of  $D = 0.023 \times 10^{-6}$  and  $0.254 \times 10^{-6} \text{ cm.}^2/\text{sec.}$  respectively. If it is assumed that the Arrhenius equation applies, the corresponding value of the energy of activation is 17.1 k.cal/mol., and the calculated value of the diffusion coefficient at 25°C. is  $2.4 \times 10^{-9} \text{ cm.}^2/\text{sec.}$  Figure 5 shows that in the moisture range 7 to 10% the value of  $D/\eta$  is 144 dynes/cm., giving for the two-dimensional resistance coefficient  $\eta = 1.67 \times 10^{-11} \text{ cm.}^2 \text{ sec/g.}$  Rouse (10) has calculated diffusion coefficients from data on the stationary state diffusion of water through films of nylon, and Bull (8) has determined the B.E.T. constants for adsorption of water by nylon.<sup>3</sup> If it is assumed that the coefficient  $\eta$

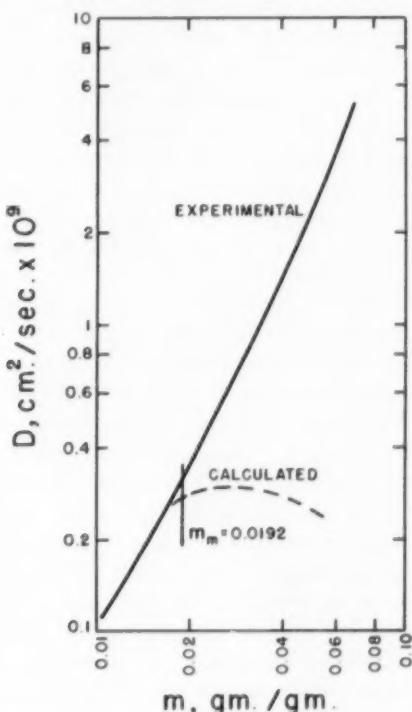


Fig. 7. The coefficient for diffusion of water through nylon as a function of moisture content, dry basis, at 25°C. The calculated curve is based on the B.E.T. expression for flow of a two-dimensional film, in which it is assumed that the resistance coefficient  $\eta$  has the same value as for wheat.

<sup>3</sup> Smith (11) has shown that there is no measurable hysteresis in the adsorption-desorption cycle of water on nylon, hence the distinction between adsorption and desorption is immaterial here.

has the same value for nylon as for wheat, then equation 22 gives a theoretical estimate of the value of the diffusion coefficient according to the B.E.T. theory. Figure 7 shows a comparison of the coefficients so calculated with those experimentally obtained by Rouse (10). The quantitative agreement at low moistures must be partly fortuitous, since the calculated diffusion coefficient for wheat is in error owing to the assumptions that the wheat kernel is spherical and is physically and chemically homogeneous, but it is a significant fact that the calculated and measured values are of comparable magnitude nearly up to the point at which multimolecular adsorption begins. At higher moistures the diffusion coefficient continues to increase with increasing moisture content, and at no point shows the type of behavior which is describable in wheat by the three-dimensional mechanism. This is probably due to the fact that nylon has a different molecular structure and has a low capacity for water sorption; at  $f = 0.95$  the equilibrium moisture content at 25°C. is only about 9%, dry basis (8).

In conclusion, the mechanisms for moisture diffusion in wheat hypothesized in this paper are in generally good accord with the salient features of the behavior of the experimentally determined diffusion coefficient. The mechanisms afford a rather definite physical picture of the process of moisture diffusion. The picture is not free from theoretical objections in some of its details and is greatly oversimplified, but should nevertheless be useful. The hypothesized mechanisms should be widely applicable to moisture diffusion in polar organic solids of natural or synthetic origin. The two-dimensional mechanism should be valid until the moisture level approaches the point of multimolecular adsorption. The present evidence suggests that the three-dimensional mechanism is applicable only at moistures greater than about 12%, dry basis, but this estimate is speculative. The chief weakness in the three-dimensional model lies in the assumptions made as to the fractional pore space, and it is suggested that the mechanism is better characterized by the energy of activation, approximately 13.8 k.cals/mol., than by the variation of the diffusion coefficient with moisture content. Energies of activation for the two-dimensional mechanism may be expected to be considerably higher. Comprehensive studies of the statics and dynamics of moisture sorption in organic solids with special regard to energies of activation and to the two-dimensional resistance coefficient  $\eta$  are required before firm generalizations are attempted.

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# EFFECT OF LEAF AND STEM RUST ON PRODUCTIVITY, DESICCATION RATE, AND KERNEL WEIGHT OF SPRING WHEAT AT SUCCESSIVE STAGES OF DEVELOPMENT<sup>1</sup>

M. N. LEVINE<sup>2</sup> AND W. F. GEDDES<sup>3</sup>

## ABSTRACT

Studies have been made on the effects of various intensities of leaf and stem rust infection respectively on susceptible varieties of spring wheat. Natural epidemics, supplemented by rust inoculations at various stages of development, were controlled to different degrees by microfine sulfur dustings at selected intervals.

Although the seasonal stem rust loads were consistently lighter than those of leaf rust, the moisture losses during maturation were accelerated much more by stem rust than by leaf rust. This was reflected in a more marked decrease in the dry-matter weight of grain per tiller and a lower average kernel weight for the plants infected with stem rust than was the case for leaf rust.

An increase of 65.0% in average leaf rust load for the years 1939-1942 decreased the average dry-matter weight of the heads by 12.5%, the grain yield by 33.0%, and the test weight by 5.4%. An increase of 31.7% in the average stem rust load for the years 1940-1942 reduced the average dry-matter weight of the heads by 24.6%, the grain yield by 45.2%, and the test weight by 11.0%.

When leaf or stem rust was prevented from attacking wheat until after the plants had reached the filling stage, the effect was as favorable as, or even more so than, when the development of either epidemic was checked at, or soon after, the plants reached the jointing stage.

Although the damage inflicted by the cereal rusts has been substantially reduced by the efforts spent in eradicating alternate hosts and in the development of resistant varieties, tremendous financial losses are caused by recurring epidemics (4, 9, 11, 13, 15, 16). Damage varies from year to year, as it depends upon a variety of biological and ecological factors; in seasons of severe rust epidemics, grain yields are sharply reduced and quality greatly impaired.

Several studies have been made on the damage caused by stem and leaf rust of wheat. In a study of the relation between stem rust infection and the yield of Marquis wheat, Goulden and Greaney (6) found that uniform increases in infection resulted in uniform decreases in

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yield. Greaney, Woodward, and Whiteside (7) showed that stem rust, alone or in conjunction with leaf rust, markedly affected yield, kernel weight, and commercial grade. When leaf rust was not a complicating factor, an increase of stem rust severity from 5 to 90% reduced the yield by 84% and the kernel weight by 65%. The test weight per bushel was reduced by 14 lb. and, in consequence, the commercial grade was lowered from "Manitoba No. 1 Hard" to "Feed Wheat." Yield and quality were inversely proportional to the percentage of rust infection.

The greenhouse and field studies carried out by Johnston (10) showed that severe leaf rust infection reduced the yield of the susceptible variety Malakof (C.I. 4898) by 55.7% and that of the resistant variety Fulhard (C.I. 8257) by 22.3%. While he observed a reduction in the size of the kernels, he noted no shriveling of the grain. Caldwell, Kraybill, Sullivan, and Compton (3) reported that, as a result of severe leaf rust infection, susceptible varieties suffered a reduction in yield ranging from 14.8% to 28.4%. They ascribed three-fourths of the losses to a reduction in the average number of kernels per head, and one-fourth to the reduction in the average kernel weight. They did not find any shriveling of kernels even under maximum leaf rust epidemic conditions. Peturson, Newton, and Whiteside (14) discovered that leaf rust infection ranging from 78 to 87% caused a reduction of 40% in yield, 27% in kernel weight, a 3.5-lb. loss in test weight, and a decrease in the commercial grade of the harvested grain.

Since 1935, studies have been in progress at the Minnesota Agricultural Experiment Station to determine the precise effects of leaf and stem rust on the productivity and quality of spring wheat and the value of chemotherapeutic fungicides as a means of controlling these fungi. The experiments and major findings with respect to productivity, desiccation, and mean kernel weight for the years 1935 to 1942 are presented in this paper.

#### Materials and Methods

Prior to 1940, the methods of treatment and the materials used varied during the several growing seasons. Thus, three common and three durum varieties were used in the 1935 tests. Microfine sulfur dust during each application was used at the rate of 50 lb. per acre to regulate the severity of the naturally occurring and artificially induced epidemics of leaf and stem rust. One series of plots received five weekly applications of sulfur; the other series received three such applications at alternate weeks; and the third series received none at all. Neither

leaf nor stem rust developed in 1936 and 1937 plots, and usable data were not obtained.

One durum and ten common wheats were involved in the 1938 study. One series received five sulfur dustings, applied at weekly intervals to the plants; in a second series, sulfur was dusted on the ground only; neither the plants nor the ground were sulfur-dusted in the third series. The stem rust and leaf rust epidemics that prevailed that year were of natural origin.

The 1939 tests were confined to a study of the effect of leaf rust. Only the variety Thatcher (C.I. 10003), theretofore and for some years afterwards outstandingly resistant to stem rust, was used in this experiment. A composite of races 5, 9, 11, 15, 28, and 65 of leaf rust was used to induce an artificial epidemic. Its intensity was regulated in different ways by means of sulfur dusting. Several of the plots received four rust inoculations, starting at the tillering stage, and no sulfur dusting whatever. Another series was similarly inoculated but in addition received four sulfur dustings on the ground. Three other series received varying numbers of rust inoculations, followed by or interspersed with varying numbers of sulfur dustings on the plants, or on the ground. Finally, one series received as many as ten sulfur dustings on the plants but no rust inoculations.

Starting with the year 1940, and continuing through 1942, a more consistently uniform method of treatments was followed. Thatcher (C.I. 10003) continued to be the variety used for determining the effects of leaf rust. Ceres (C.I. 6900) or Marquis (C.I. 3641) served as test varieties for studying the effects of stem rust, even though subsequently both were reported to be susceptible also to leaf rust (12). Epidemics were induced by artificial inoculations with uredial composites of available physiologic races. The intensities of the respective rust epidemics were regulated by varying numbers of sulfur dustings applied at the rate of 50 lb. per acre. Two methods of control, as described by Hoffer, Geddes, and Levine (8), were used. By the so-called preventive method, the incidence of rust was delayed until the desired stages in plant development were reached; after this, artificial rust inoculations were made at weekly intervals. By the inhibitive method, rust infection was induced in the early stages of plant growth; further progress of the respective epidemics was arrested at the desired periods by weekly applications of sulfur dust.

There were altogether six variations of the treatments used, namely: 1) Eight applications of sulfur onto the developing wheat plants from the tillering to the fruiting stages, with no artificial rust inoculations being used at any time; 2) six applications of sulfur on the plants,

followed or preceded by two inoculations of rust; 3) four applications of sulfur on the plants, followed or preceded by four rust inoculations; 4) two applications of sulfur on the plants, followed or preceded by six rust inoculations; 5) no sulfur dusting applied to either plants or ground, but plants receiving eight rust inoculations beginning with the tillering stage and continuing to the fruiting stage; 6) eight rust inoculations made during the identical plant development stages as above, each inoculation preceded or followed by an application of sulfur dust to the ground only.

Observations according to the modified "Cobb scale" of rust estimates were made at the following eight kernel formation stages: pre-milk, early milk, late-milk, soft-dough, medium-dough, hard-dough, semi-ripe, and full ripe, unless otherwise specified.

The methods described by Geddes and Levine (5) for obtaining and preparing samples for thiamine determinations were used in this study for the determination of moisture losses and dry-matter weights. A week after flowering, when the heads were in the pre-milk stage, and semiweekly thereafter, a certain number of entire aerial tillers were collected and weighed, after which the heads were clipped off and weighed. These two major fractions were then dried at approximately 60°C. and their dry weights recorded. After threshing of the heads, the air-dry weights of the kernels and of the nonkernel portion were determined. In this manner a total of three plant fractions was obtained, namely: grain (kernels), chaff (glumes and rachides), and straw (stems and leaves). Each of these fractions was ground in a Wiley laboratory mill to pass a sieve having 0.5-mm. openings, and stored in air-tight containers.

The moisture contents of the air-dried samples were determined by the 1-hour, 130°C. air-oven method (1). The moisture present in the entire tillers at each stage of maturity was computed from the weights of the various fractions and of the moisture lost on air-drying and oven-drying. As an additional index of maturity and also as a measure of the influence of rust infection on the filling of the kernels, the dry-matter weights per thousand kernels were determined. Five hundred air-dry kernels were counted and weighed in duplicate and the results computed to a dry-matter basis.

As the wheat reached full maturity, the center row of each test plot was flooded with water and the plants pulled out with their roots intact. After they were thoroughly washed and air-dried, the roots were cut off below the basal node and weighed to ascertain what effect different severities of rust infection may have had on root development.

### Results

1935. The initial studies in 1935 involved the application of sulfur dust to three varieties each of common and of durum wheat. The mean results for the untreated control and for two series of dusting treatments for each class of wheat are recorded in Table I. The five continuous weekly sulfur dustings reduced more effectively the severity of leaf rust infection on the common wheats than they did that of stem rust; the reverse was true of the durum varieties. The common wheats suffered a mean loss of 28.5% in yield and 17.8% in kernel weight; the corresponding losses for the durum varieties were 22.2 and 18.0% respectively. Where only three intermittent sulfur dusting were used, the results were somewhat intermediate between the two conditions discussed above, insofar as the common wheats were concerned. This was not uniformly so with the durum varieties, particularly as regards the severity of stem rust infection and kernel weight.

TABLE I  
EFFECT OF LEAF AND STEM RUST INFECTION ON THE YIELD AND KERNEL WEIGHT OF COMMON AND DURUM WHEATS. MEAN RESULTS FOR 1935

FACTORS CONSIDERED	COMMON WHEATS <sup>a</sup>		DURUM WHEATS <sup>b</sup>		
	Untreated	Number of Dustings with Sulfur	Untreated	Number of Dustings with Sulfur	
	3	5		3	5
Stem rust, %	55.0	45.0	36.7	33.3	33.3
Leaf rust, %	41.7	25.0	13.3	11.7	8.3
Yield, bu/acre	20.3	24.0	28.4	31.6	34.1
Kernel weight, mg.	19.4	20.4	23.6	26.4	25.9

<sup>a</sup> Data are the mean values for Kota (C.I. 5078), Marquis (C.I. 3641), and Reliance (C.I. 7370).

<sup>b</sup> Data are the means for Acme (C.I. 5284), Kubanka (C.I. 1440) and Mindum (C.I. 5296).

1938. Although the results obtained in 1938 were generally inconclusive, notable effects of sulfur dusting were obtained with two of the ten common varieties included in the test. The treated plots of Ceres (C.I. 6900) averaged 68.0% stem rust and 25.0% leaf rust, compared with 85.0% stem rust and 80.0% leaf rust in the untreated plots. The former yielded 10.8 bu. per acre as against 5.5 in the latter, thus showing a gain of 96.4% for the treated plots. In the case of Marquis (C.I. 3641), the sulfur treatments appeared to have produced absolutely no effect on the severity of stem rust, the average infection in either case being 95.0%. However, the 20.0% leaf rust in the treated plots was in sharp contrast to the 75.0% in the untreated plots. The difference in yield between 9.8 and 7.3 bu. per acre constituted a gain of 34.2%. The one durum variety included in the test, Mindum (C.I. 5296), was somewhat susceptible to stem rust and nearly immune from leaf rust and remained unaffected by the sulfur treatments as far as the severity of

infection was concerned. But, for some unknown reason, its yield was adversely affected, in comparison with the untreated plots, by the sulfur dust applied to the plants and favorably affected when the ground only was dusted.

1939. Very marked results were obtained with sulfur treatments against leaf rust in 1939. The six treatments used that year produced significant decreases in the severity of infection. The increases in yield and quality were also quite pronounced. Where the plants were sulfur-dusted ten times, the average severity of leaf rust infection was 17.5%, compared with 98.8% where they were not dusted at all. The resulting effects were as follows: yield, 21.7 and 12.5 bu. per acre; test weight, 58.0 and 52.8 lb. per bu.; average kernel weight, 19.3 and 15.6 mg.; market value of crop, \$19.51 and \$10.53 per acre, respectively. An analysis of variance revealed that the differences between treatments were significant. Calculating the regression coefficient and determining the regression equation for the results obtained, it was found that 1% of leaf rust caused a yield reduction of 0.1 bu. per acre. Thus, each 10% of leaf rust reduced the yield of the Thatcher (C.I. 10003) wheat tested in 1939 by 4.6%.

1940 and 1941. The study of the effect of leaf and stem rust on moisture content and dry matter weight at successive stages of kernel formation was limited to the years 1940 and 1941. A composite of the data for the effects of the lightest and heaviest infections is given in Table II.

In the case of eight applications of sulfur and no rust inoculations, the leaf rust infection increased from 7.1% at the pre-milk stage to 35.3% by the time the plants were dead ripe; thus carrying an average seasonal rust load of 20.3%. When the plants received eight weekly leaf rust inoculations and no sulfur dust, the severity of infection increased from 73.8 to 99.0% as the plants matured (average seasonal load 88.5%). At the pre-milk stage, the moisture contents of the heads and tillers, the dry-matter weights of the grain, and the average kernel weights for the lightly and heavily rusted plants were quite similar. However, as the wheat matured, there was a more rapid loss of moisture from the heavily rusted plants, a smaller increase in the dry-matter weight of the grain and of the entire tiller (aerial portion), and a lower increase in average kernel weight than was observed for the lightly rusted plants. Thus, the more rapid loss of moisture from the heavily rusted tillers resulted in a moisture content 12.1% lower than that of the lightly rusted plants at the semiripe stage. The more rapid loss of moisture from the heavily leaf-rusted plants provided less opportunity for photosynthesis and for the translocation of nutrients into

TABLE II  
EFFECT OF SUCCESSIVE STAGES OF KERNEL DEVELOPMENT ON MOISTURE CONTENT, DRY-MATTER WEIGHT, AND AVERAGE KERNEL WEIGHT  
OF HARD RED SPRING WHEAT VARIOUSLY AFFECTION BY CERAL RUST  
(Mean results for 1940 and 1941)

Days after Bloom	Successive Development Stages	Rust Infect- ion	Moisture Content			Dry Matter Weight per Tiller			Man- Kernel Weight mg.	Moisture Content			Dry Matter Weight per Tiller			Man- Kernel Weight mg.	
			Heads		Tillers	Grain		Tillers		Heads		Tillers	Grain		Tillers		
			%	%	%	%	%	%		%	%	%	%	%	%		
Seasonal Average Leaf Rust Load — 20.3%																	
7	Pre-milk	7.1	68.7	69.6	11.1	167.8	4.3	73.8	68.9	69.3	11.7	157.9	4.5				
10	Early milk	9.4	67.1	68.5	23.8	176.3	7.6	78.6	66.7	67.5	22.3	158.7	7.7				
14	Late milk	12.9	61.7	64.9	39.4	194.0	11.2	81.6	59.7	62.9	34.0	166.3	11.3				
17	Soft dough	18.5	58.2	62.9	51.0	195.6	14.8	90.3	56.8	61.4	42.9	166.2	14.4				
21	Medium dough	23.0	50.9	57.2	65.8	200.5	19.6	92.0	49.3	54.5	56.8	176.9	18.4				
24	Hard dough	28.0	46.2	52.5	70.8	198.1	22.5	96.0	42.9	47.9	58.0	171.3	19.8				
28	Semi-ripe	29.0	35.5	44.3	65.7	181.8	23.3	97.1	26.5	32.2	57.6	164.4	20.0				
31	Dead ripe	35.3	27.1	35.2	64.3	177.6	23.7	99.0	24.2	26.4	54.6	154.0	20.4				
Seasonal Average Stem Rust Load — 3.9%																	
7	Pre-milk	1.0	67.7	67.2	14.9	192.7	5.9	28.8	66.3	65.9	14.9	177.3	5.5				
10	Early milk	2.1	65.6	65.2	29.5	201.7	9.9	35.8	65.4	64.8	25.1	180.5	9.1				
14	Late milk	2.9	59.0	60.9	43.9	190.9	14.5	49.9	55.2	59.4	36.6	176.8	12.8				
17	Soft dough	3.8	53.2	55.6	56.6	203.7	18.6	53.2	52.2	53.3	37.7	170.9	14.3				
21	Medium dough	5.0	44.2	47.4	67.4	207.5	22.0	61.0	29.4	32.1	36.5	159.9	14.8				
24	Hard dough	5.8	31.9	36.8	72.9	211.4	23.2	65.8	17.4	18.2	40.8	163.0	15.2				
28	Near ripe	6.5	20.8	23.3	70.6	198.5	23.2	71.0	18.1	19.0	43.0	168.6	16.0				

the grain; as a result, the increase in dry-matter weight of the grain per tiller from the pre-milk to the dead-ripe stage was only 42.9 cg. as compared with 53.2 cg. for the less severely leaf-rusted plants. The corresponding increases in average kernel weight were 15.9 and 19.4 mg. respectively.

During the two years 1940 and 1941, the average stem rust load on the plants receiving eight applications of sulfur dust and no rust inoculations amounted to 3.9%, whereas the infection caused by the plants' receiving eight inoculations and no sulfur treatments averaged 52.2%. As in the case of leaf rust, the more severely infected plants lost moisture more rapidly and showed smaller increases in dry-matter weight and average kernel weight with progressive maturity than the lightly stem-rusted plants. The effect of a heavy stem rust load on these properties was more pronounced than that of leaf rust, even though the average load was much lower (52.2% vs. 88.5%). The more rapid desiccation of the heavily stem-rusted plants resulted in a moisture content of the tillers which was lower by as much as 18.6% than was found at the corresponding stage (hard-dough) in the lightly infected plants. The increase in dry-matter weight of the grain per tiller from the pre-milk to the near-ripe stage was only 28.1 cg. as compared with 55.9 cg. for those lightly infected with stem rust. The corresponding increases in average kernel weight were 10.5 and 17.3 mg. respectively.

A concise summary of the data bearing on the relative merits of the preventive and inhibitive methods used to regulate the inception or termination of stem and leaf rust epidemics is presented in Table III. It covers the 3-year period 1940-1942, and deals only with the epidemic effects on the jointing, heading, and filling stages. It is fortunate that during this time leaf rust was not a complicating factor on either Ceres or Marquis.

Postponing the inception of the leaf rust epidemic until the filling stage reduced average rust load by 62.8%, increased yield by 7.6 bu/acre, improved test weight by 3.0 lb/bu., and kernel weight by 2.9 mg.; advancing its termination to the jointing stage reduced average rust load by 61.5%, increased yield by 7.6 bu/acre, improved test weight by 2.9 lb/bu., and kernel weight by 3.9 mg. Similarly, postponing the inception of the stem rust epidemic until the filling stage reduced average rust load by 34.3%, increased yield by 9.0 bu/acre, improved test weight by 6.9 lb/bu., and kernel weight by 5.7 mg.; advancing its termination to the jointing stage reduced average rust load by 27.9%, increased yield by 7.4 bu/acre, improved test weight by 5.2 lb/bu., and kernel weight by 5.2 mg. Thus, preventing the develop-

## EFFECTS OF RUST ON SPRING WHEAT

TABLE III  
RELATIVE EFFECTS OF TIME OF INCEPTION AND TERMINATION OF LEAF AND STEM RUST EPIDEMICS OCCURRING AT THREE DEVELOPMENTAL STAGES OF THE PLANT ON THE YIELD, TEST WEIGHT, AND AVERAGE KERNEL WEIGHT OF HARD RED SPRING WHEAT  
(Mean results for the years 1940, 1941 and 1942)

FACTOR CONSIDERED	LEAF RUST						STEM RUST					
	Inception at Given Stages			Termination at Given Stages			Inception at Given Stages			Termination at Given Stages		
	Joining	Heading	Filling	Jointing	Heading	Filling	Jointing	Heading	Filling	Jointing	Heading	Filling
Seasonal rust load, %												
1940	87.1	42.5	8.8	13.3	44.2	95.4	59.4	30.0	5.1	4.3	19.8	51.5
1941	89.2	74.0	26.9	36.5	58.8	89.8	45.8	12.8	2.2	3.5	6.2	12.0
1942	74.6	42.9	26.8	34.4	62.5	83.7	10.4	12.1	5.4	3.5	6.2	12.0
Annual mean infection												
Yield, bu/acre	83.6	53.1	20.8	28.1	55.2	89.6	38.5	18.3	4.2	3.9	13.0	31.8
1940	26.5	31.4	32.0	27.0	27.8	29.9	10.4	14.6	26.7	22.9	16.4	8.5
1941	14.6	17.0	26.6	26.6	23.0	19.0	11.5	17.4	21.1	12.2	10.0	10.3
1942	11.0	15.6	16.4	15.4	11.7	9.3	11.2	11.1	12.2	10.0	10.3	9.4
Annual mean yield												
Test weight, lb/bu	17.4	21.3	25.0	23.0	20.8	15.4	11.0	14.4	20.0	16.4	13.4	9.0
1940	59.0	60.0	60.5	60.4	59.4	57.4	43.0	45.2	56.8	57.2	59.9	57.6
1941	53.6	57.2	60.4	59.9	59.3	56.4	52.8	56.2	57.2	57.3	55.6	54.0
1942	54.9	55.6	57.6	56.8	56.6	54.6	54.8	55.6	57.3	55.6	54.4	51.1
Annual mean test weight												
Average kernel weight, mg.	56.3	57.6	39.5	59.0	58.4	56.1	50.2	52.3	57.1	57.8	56.0	52.6
1940	23.5	26.1	26.6	26.2	23.8	20.7	12.1	13.1	21.2	25.3	27.8	24.8
1941	19.7	19.8	23.4	23.9	23.4	19.6	19.5	23.2	25.3	27.8	16.3	15.2
1942	16.1	16.4	18.0	16.3	16.0	14.4	17.7	17.7	18.9	16.3	16.2	15.8
Annual mean kernel weight	19.8	20.8	22.7	22.1	21.1	18.2	16.1	18.0	21.8	22.0	20.5	16.8

COMPARATIVE EFFECTS OF CHEMICALLY CONTROLLED AND ARTIFICIALLY INDUCED CEREAL RUST EPIDEMICS ON THE PRODUCTIVITY, PHYSICAL CHARACTERISTICS, AND COMMERCIAL VALUE OF HARD RID SPRING WHEAT

(Data for leaf rust are mean results for 1939-1942; data for stem rust are means for 1940-1942)

FACTORS CONSIDERED	LEAF RUST			STEM RUST		
	Infection Meager	Infection Severe	Deviation Amount	Percent	Infection Slight	Deviation Amount
Severity of seasonal rust load, <sup>a</sup> %	22.5	87.5	+65.0		4.1	35.8
Transferred rust load percentage values, %	28.3	69.3	+41.0	+144.9	11.7	36.8
Length of headed tillers, cm.	80.8	76.1	-4.7	-5.8	87.2	84.3
Weight of headed tillers, dg.	15.9	13.1	-2.8	-17.6	17.0	14.4
Weight of roots, dg.	1.2	0.8	-0.4	-33.3	1.4	1.0
Weight of stems, dg.	9.1	7.4	-1.7	-18.7	9.5	8.8
Weight of heads, dg.	5.6	4.9	-0.7	-12.5	6.1	4.6
Weight of kernels, mg.	21.5	18.0	-3.5	-16.3	21.2	15.9
Weight of good kernels, mg.	24.4	20.8	-3.6	-14.8	25.6	18.0
Weight of poor kernels, mg.	11.6	10.9	-0.7	-6.0	12.3	10.2
Test weight of harvested grain, lb/bu.	58.8	55.6	-3.2	-5.4	57.4	51.1
Test weight of good kernels, lb/bu.	61.0	57.6	-3.4	-5.6	59.6	54.4
Test weight of poor kernels, lb/bu.	51.1	48.9	-2.2	-4.3	50.3	48.1
Estimated yield, bu/acre	23.0	15.4	-7.6	-33.0	18.6	10.2
Market value per acre of harvested crop, \$	21.90	13.84	-8.06	-36.8	18.85	10.07
					18.85	-8.78
						-46.6

ment of either epidemic past the heading stage or arresting it prior to this stage produced very favorable results.

The data recorded in Table IV represent the mean results obtained for leaf rust during the four years, 1939-1942, and for stem rust during the three years 1940-1942. Only the effects of the most and least severe infections are considered. In addition to the percentage values of the average seasonal rust loads, their transferred values, as determined by the methods described by Bliss (2), are also indicated in the table.

An increase of 65.0% in the 4-year average severity of the leaf rust load reduced the average length of the headed tillers by 5.8% and their average weight by 17.6%. The reduction in the average weight of the roots amounted to 33.3%, of the stems 18.7%, and of the heads 12.5%. The average weight of the kernels was reduced by 16.3% and the test weight of the harvested grain as a whole was lowered by 5.4%. The yield losses averaged 33.0%, while the depreciation in the monetary value of the harvested crop amounted to 36.8%.

An increase of 31.7% in the three-year average severity of the stem rust load reduced the length of the headed tillers by 3.3% and their weight by 15.3%. The root weight was reduced by 28.6%, that of the stems by 7.4%, and of the heads by 24.6%. The normal kernels were lighter by 25.0%, and the test weight of the harvested grain in general was lowered by 11.0%. The yield losses averaged 45.2%, whereas the commercial devaluation of the harvested crop amounted to 46.6%.

The critical period in the development of leaf rust epidemics seems to be somewhere between the booting and heading stages, while that of stem rust epidemics seems to be somewhere between the heading and flowering stages. The results thus far obtained indicate that the fate of a wheat crop may depend on whether a rust epidemic was permitted to establish itself prior to the heading stage or was not interfered with in its further development thereafter.

These studies show that moderately severe stem rust epidemics are likely to inflict greater injury to susceptible spring wheat varieties than very severe leaf rust epidemics. In the case of Ceres or Marquis wheat, an average stem rust load of 35.8% reduced the yield by 8.4 bu/acre, while the reduction in yield caused by an average leaf rust load of 87.5% on Thatcher wheat amounted to 7.6 bu/acre. In either case there was a test weight debasement of 2.2 lb/bu. Stem rust reduced the average kernel weight by 5.3 mg., leaf rust by 3.5 mg. While the monetary acre loss of \$8.78 caused by stem rust was only slightly more than the \$8.06 caused by leaf rust, percentagewise these losses amounted to 46.6% and 36.8% respectively.

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## MOISTURE RELATIONS IN WHEAT AND CORN<sup>1</sup>

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### ABSTRACT

Hysteresis loops were established for wheat and corn at 25°, 30°, and 35°C. over the range 0 to 97% relative humidity. Both desorption and adsorption isotherms for corn and wheat were found to be sigmoid. The maximum hysteresis effect was found between 12 and 44% humidity amounting to 1.6% and diminishing to less than 0.2% at 92% humidity.

No consistent differences were found in the extent of hysteresis with temperature changes over the range of temperatures studied. Hygroscopic capacity of grain shows a negative correlation with temperature, a rise in temperature of 10°C. lowering the equilibrium moisture content by as much as 1.3%.

The equilibrium relative humidity of the interseed air as measured with an electric hygrometer and the moisture content as measured with an electric moisture meter were determined on 114 commercial samples of wheat and corn. Values found were shown to lie, for the most part, in the expected range of the hysteresis loops established.

Observations were made on the relative importance of moisture content of grain or relative humidity of interseed air near the critical levels for mold growth.

The importance of low moisture content for safe storage of farm commodities has been known for many years. The close relationship between relative humidity of storage air and the moisture content of stored products also has been well recognized. In recent years many workers have studied these relationships from different standpoints, using various methods and many varieties and classes of grain and other farm commodities. Much of the information on cereal grains has been well summarized in the recent monograph edited by Anderson and Alcock (1), and its extensive bibliographies serve as a reference source for detailed studies of specific phases of storage problems. Some of these workers (6, 7, 8, 10) report hygroscopic capacities of grain at different relative humidities and temperatures without regard to hysteresis effects. Others (9, 12, 14) report average figures for samples desorbing and adsorbing moisture. Only Babbitt (2, 3) has plotted the hysteresis effect in moisture sorption of wheat over the full range from 0 to 100% relative humidity. Hysteresis data on rough rice (4) and on polished rice (13) have been reported.

Most materials of plant origin exhibit hysteresis in the adsorption and desorption of water. Sigmoid curves generally have been reported where there is no marked blocking of water movement such as is found

<sup>1</sup> Manuscript received January 29, 1957. Contribution from the Northern Utilization Research and Development Division, Peoria, Illinois, one of the divisions of the Agricultural Research Service, U. S. Department of Agriculture.

in the "hard seed" condition typical of some legume seeds. Babbitt reports sigmoid curves for desorption, but, in his earlier study (2), reported an adsorption curve distinctly convex to the relative humidity axis. Even though some of the data in his later experiments indicated a possible sigmoid adsorption curve, he concludes from the preponderance of his data that the adsorption curve is not sigmoid, but convex.

The studies here reported were undertaken to extend our knowledge of the relationship between relative humidity of storage air and moisture content of stored grain by establishing complete desorption and adsorption isotherms for varieties representative of the major classes of wheat and corn, and to compare these data with values found for commercial samples.

### Materials and Methods

Experiments were made to determine the percent moisture in wheat and corn at equilibrium with air of a series of relative humidities from 0 to 92%. The desired humidities were maintained in 6-in. desiccators by means of saturated salt solutions (5, 11, 16, 17) (Table I).

TABLE I  
SATURATED SALT SOLUTIONS USED AND RELATIVE HUMIDITIES REPORTED

SALT	RELATIVE HUMIDITY			REFERENCE
	25°C. %	30°C. %	35°C. %	
K <sub>2</sub> SO <sub>4</sub>	96.9	96.6	96.4	16
KNO <sub>3</sub>	92.0	90.7	89.3	16
KCl	84.7	84.5	83.0	5
NH <sub>4</sub> Cl	77.0	78.2	76.6	11
NaCl	75.8	75.6	75.5	16
NaNO <sub>3</sub>	73.2	72.8	72.1	5
Na <sub>2</sub> CrO <sub>4</sub>	66.0	64.6	63.2	5
NaBr	57.0	56.3	54.6	17
K <sub>2</sub> CO <sub>3</sub> ·2 H <sub>2</sub> O	43.8	43.5	43.4	17
MgCl <sub>2</sub> ·6 H <sub>2</sub> O	33.2	32.8	32.5	16
KC <sub>2</sub> H <sub>3</sub> O <sub>2</sub>	22.6	22.0	21.0	17
LiCl·H <sub>2</sub> O	12.0	11.8	11.7	16
P <sub>2</sub> O <sub>5</sub> or vacuum oven 78°C.	0.0	0.0	0.0	17

Desiccators were evacuated to the approximate vapor pressure of the respective salt solutions to accelerate moisture transfer. The small aluminum moisture dishes used did not corrode or change weight if care was taken to prevent boiling of the salt solution during evacuation. Temperatures were controlled to  $\pm 0.15^{\circ}\text{C}$ . by complete immersion of the desiccators in a water bath. Samples were stored at 5°C., 60–64% humidity, from time of collection until studied.

Gane (7) reports that coarsely ground wheat gives hygroscopic values essentially equal to those of whole-kernel wheat and in much less time. After confirmation of this observation on one sample of wheat, most of these studies were made on grain ground to pass 20-mesh with an Intermediate Model Wiley Mill using replicated 2-g. samples. Ten-grain replicates of one sample each of whole-kernel wheat and corn are also included.

The basic moisture method was that prescribed for wheat in S.R.A. No. 147 (15) and consisted of drying replicated 2-g. ground samples in a triple-wall oven for 1 hour after the oven had returned to 130°C. after insertion of the samples. This same method was used for the corn rather than the 96-hour water-oven method officially prescribed for whole corn, since most moisture sorption data were taken on 2-g. aliquots of the ground grain. All moisture data are reported as percent of total weight calculated from weight changes, assuming dry matter to remain constant. Data reported represent averages of no less than two replicates, four or six replicates being used for most of the 25° and 30°C. data. "Zero moisture content" as the starting point for the adsorption curves was attained by drying samples in a vacuum oven at 72–76°C. for 3 days or by drying over phosphorus pentoxide to constant weight. On the ground samples these methods agreed with the 130°C. oven method within 0.2% moisture. Starting point for the desorption curves was around 23% moisture, attained by exposing samples to an atmosphere of 97% humidity overnight.

Most data were obtained from samples moved stepwise around the complete adsorption-desorption cycle. Considerable time was saved by shifting samples by 20% humidity steps, data being taken at alternate levels on half the samples and at the intervening levels on the others. Time allowed for attainment of equilibrium varied somewhat, depending particularly on humidity level and also on whether the samples were desorbing or adsorbing moisture. Time required for the ground samples was as follows: above 75% R.H., 2–4 days; 32–75%, 4–7 days; 22%, 7–12 days; and 11%, 12–15 days. Preliminary data indicated that these periods gave results within 0.1% moisture of similar samples held as much as three times as long.

Equilibrium relative humidities of a number of samples of whole grain were measured at 30°C. with an Aminco-Dunmore<sup>2</sup> hygrometer, found accurate to  $\pm 1.5\%$  humidity. Grains studied included, from Illinois, 60 samples of wheat (predominantly hard red winter) and 38 samples of yellow dent corn. Thirteen hard red spring and three durum

<sup>2</sup> Mention of trade names or equipment does not constitute endorsement by the U. S. Department of Agriculture over others of a similar nature not mentioned.

wheat samples from Minnesota also were studied. All were held in moistureproof cans at 25°C. until equilibrium humidities could be determined. Moisture contents of these were determined with the Tag-Heppenstall moisture meter. Humidity determinations were carried out as follows: Approximately 60 g. of grain were placed in a 150-ml. extraction flask immersed to the neck in a constant-temperature water bath. A single humidity- and temperature-sensing element (selected to cover the expected range of humidity as indicated by the moisture test) was inserted through a rubber stopper just to the surface of the grain and the equilibrium humidity determined when a constant reading had been maintained for 1 hour. Total time for most samples to attain equilibrium was 2 to 4 hours.

### Results and Discussion

*Effects of Temperature on Hysteresis.* Hysteresis loops were established for varieties representative of the wheat classes hard red winter, hard red spring, durum, and white wheat and yellow dent corn at 25°, 30°, and 35°C. In contrast to the only previously published complete loops on wheat (2, 3), both desorption and adsorption isotherms were found to be sigmoid (Fig. 1). The maximum hysteresis effect occurred between 12 and 44% humidity and amounted to 1.6% moisture. Above

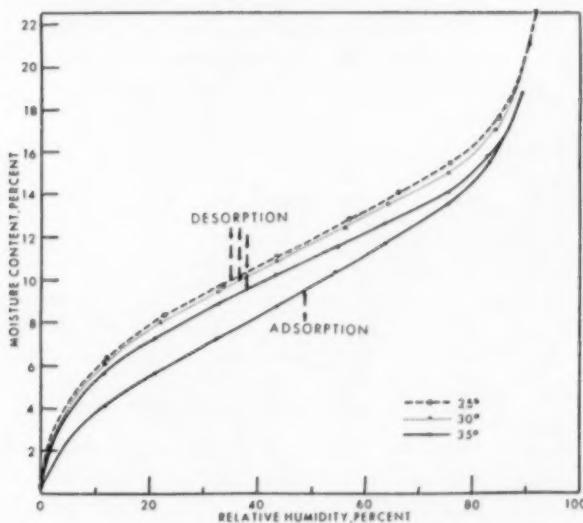


Fig. 1. Hysteresis in moisture sorption in wheat. Desorption isotherms at 25°, 30°, and 35°C. Adsorption isotherm at 35°C.

56% humidity the hysteresis effect diminished gradually to less than 0.2% moisture at 92% humidity.

Moisture content of grain in equilibrium with nine humidities at 30°C. is shown in Table II. A typical hysteresis loop is shown in Fig. 1

TABLE II  
HYGROSCOPIC MOISTURE IN WHEAT AND CORN AT 30°C.

VARIETY	RELATIVE HUMIDITY								
	Moisture Content								
	11.8	22.0	32.8	43.5	53.3	64.6	75.6	84.5	90.7
Wheat — Desorption									
Elgin	6.20	8.20	9.70	11.06	12.63	13.98	15.52 <sup>c</sup>	17.59	21.56
Stewart	5.78	7.66	9.17	10.56	12.18	13.34	14.87	16.94	21.34
Pawnee <sup>a</sup>	6.15	8.16	9.65	10.99	12.28	13.16	14.69 <sup>b</sup>	16.43	20.59
Pawnee <sup>b</sup>	5.98	8.03	9.56	11.05	12.39	13.41	14.52	16.94	20.70
Pawnee <sup>c</sup>	6.12	8.36	9.63	11.02	12.58	13.87	15.32	17.21	
Pawnee <sup>d</sup>	6.05	8.05	9.50	10.83	12.40	13.68	15.18 <sup>a</sup>	17.18	21.00
Mida	6.08	7.96	9.45	10.89	12.46	13.68	15.12	17.15	21.45
Average	6.05	8.06	9.52	10.91	12.42	13.59	15.03	17.06	21.11
Wheat — Adsorption									
Elgin	4.87	6.73	8.12	9.61	11.39	12.94	14.58 <sup>c</sup>	17.03	21.44
Stewart	4.19	6.07	7.67	9.25	10.90	12.65	14.11	16.71	21.11
Pawnee <sup>a</sup>	4.84	6.76	8.21	9.76	11.41	12.83	14.55 <sup>b</sup>	16.80	21.19
Pawnee <sup>b</sup>	4.69	6.48	8.20	9.52	11.25	12.70	14.52	16.69	20.78
Pawnee <sup>c</sup>	4.47	6.46	8.01	9.46	11.29	12.68	14.40		
Pawnee <sup>d</sup>	4.42	6.41	7.79	9.26	11.05	12.55	14.12 <sup>a</sup>	16.57	20.76
Mida	4.50	6.29	7.91	9.45	11.10	12.78	14.36	16.84	21.34
Average	4.57	6.46	7.99	9.47	11.20	12.73	14.38	16.77	21.10
Av. diff. (desorption — adsorption)	1.48	1.60	1.53	1.44	1.22	0.86	0.65	0.26	0.01
Corn — Desorption									
Schwenk 13 <sup>e</sup>	5.66	7.72	9.26	10.73	12.46	13.82	15.28	16.99	20.21
Schwenk 13 <sup>f</sup>	6.57	8.30	9.88	11.19	12.67	14.02	15.50 <sup>d</sup>	17.21	20.65
Dyar 444	5.78	7.59	8.99	10.28	11.85	13.17	14.75 <sup>b</sup>	16.90	20.30
Illinois 1277	5.85	7.65	9.07	10.30	11.91	13.12	14.61	16.38	19.66
Average	6.07	7.85	9.31	10.59	12.14	13.43	14.95	16.83	20.20
Corn — Adsorption									
Schwenk 13 <sup>e</sup>	4.39	6.17	7.66	9.06	10.93	12.25	14.17	16.13	19.56
Schwenk 13 <sup>f</sup>	5.11	7.17	8.38	10.20	11.48	12.81	14.56 <sup>d</sup>	16.14	20.54
Dyar 444	4.45	6.17	7.58	9.04	10.49	11.91	13.71 <sup>b</sup>	15.90	19.69
Illinois 1277	4.88	6.56	7.79	9.16	10.68	11.95	13.56	15.62	19.74
Average	4.81	6.67	7.92	9.37	10.88	12.22	13.94	15.89	19.99
Av. diff. (desorption — adsorption)	1.26	1.18	1.39	1.22	1.26	1.21	1.01	0.94	0.21

<sup>a</sup> 1952 Crop from Illinois, ground; 20-mesh.

<sup>b</sup> 1952 Crop from Illinois, sound, whole kernels.

<sup>c</sup> 1953 Crop from Illinois, ground; 20-mesh.

<sup>d</sup> 1952 Crop from Nebraska, ground; 20-mesh.

<sup>e</sup> 1953 Crop, sound, whole kernels.

<sup>f</sup> 1953 Crop, ground; 20-mesh.

A, B, C, D, E, hysteresis redetermined at 75.6% relative humidity after 1-2 years' storage (Table IV).

TABLE III  
HYSTERESIS AT 25°, 30°, AND 35°C.

TEMPERATURE (°C.)	APPROXIMATE RELATIVE HUMIDITY *								
	DIFFERENCE BETWEEN AVERAGE DESORPTION AND ABSORPTION MOISTURE CONTENT								
	12	22	33	44	56	65	76	84	91
Wheat									
25°	1.45	1.49	1.47	1.28	1.15	0.97	0.79	0.34	0.26
30°	1.48	1.60	1.53	1.44	1.22	0.86	0.65	0.26	0.01
35°	1.56	1.61	1.53	1.56	1.15	0.84	0.62	0.33	...
Corn									
25°	1.40	1.52	1.46	0.94	1.01	1.05	0.55	0.52	0.19
30°	1.26	1.18	1.39	1.22	1.26	1.21	1.01	0.94	0.21
35°	1.50	1.50	1.62	1.40	1.33	1.06	0.96	0.36	0.29

\* See Table I for differences in relative humidity due to the temperature coefficient of the saturated salt solution used at each humidity level.

plotted from the averages of data at 35° C. together with desorption curves for 30° and 25°C. The extent of hysteresis at these temperatures is shown in Table III. Wheat shows no consistent differences in extent of hysteresis which may be ascribed to temperature. Curves are similar but not strictly parallel at the three temperatures studied. Data for whole-kernel and ground wheat were in very close agreement. The data for corn are more limited and also more variable. Good agreement is shown at some humidity levels, but differences up to 1% moisture between the whole-kernel and ground corn were found. It is suspected that some dry-matter losses occurred in the whole corn, possibly due to the length of time it was held at high humidity to attain equilibrium.

*Effects of Age of Sample on Hysteresis.* The extent of hysteresis at 75.6% humidity, 30°C., was determined on three wheat and two corn samples after 1 to 2 years' storage [see Table IV (A-E)] and found to be less than 50% of that found earlier at this level [Table II (A-E)] with the exception of the 1952 crop Pawnee wheat from Illinois (B). This sample showed unusually low (0.14%) hysteresis in both the earlier and later determinations. The hysteresis at this temperature and humidity was measured on a number of additional samples which had been stored for varying periods. Results (Table IV) indicate that the decrease in hysteresis noted is not a continuing result of age, but do not eliminate the possibility of some changes in hysteresis in the immediate post-harvest period.

*Hysteresis in Sorption Over a Limited Moisture Range.* The width of the hysteresis loop is determined to some extent by the humidity range to which it is exposed. In Fig. 2 the adsorption isotherms from approximately 10, 8, and 6% moisture are plotted for one lot of wheat

TABLE IV  
HYSTERESIS AND HYGROSCOPIC MOISTURE IN WHEAT AND CORN IN RELATION TO CLASS,  
CROP YEAR, AND LOCATION AT 75.6% RELATIVE HUMIDITY, 30°C.

VARIETY	CLASS	CROP YEAR	LOCATION	MOISTURE		HYSTERESIS OR DIFFERENCE
				Desorp- tion %	Absorp- tion %	
<b>Wheat</b>						
Pawnee	HRW	1941	Manhattan, Kansas	15.00	14.80	0.20
Pawnee	HRW	1946	Woodward, Oklahoma	15.20	14.96	0.24
Pawnee	HRW	1946	Lincoln, Nebraska	14.91	14.76	0.15
Pawnee	HRW	1952	Lincoln, Nebraska	15.06	14.61	0.45 <sup>a</sup>
Pawnee	HRW	1952	Peoria, Illinois	14.89	14.75	0.14 <sup>b</sup>
Elgin	Soft white	1943	Pullman, Washington	15.14	14.79	0.35
Elgin	Soft white	1952	Pullman, Washington	15.30	14.93	0.37 <sup>c</sup>
Trumbull	SRW	1951	Wooster, Ohio	15.36	15.00	0.36
<b>Corn</b>						
U.S. 13	Yellow dent	1940	Ames, Iowa	15.07	14.46	0.61
U.S. 13	Yellow dent	1950	Ames, Iowa	15.27	14.61	0.66
Schwenk 13	Yellow dent	1953	Peoria, Illinois	14.81	14.43	0.38 <sup>d</sup>
Dyar 444	Yellow dent	1954	Peoria, Illinois	14.64	14.31	0.33 <sup>e</sup>
Iowa 306	Yellow dent	1948	Ames, Iowa	14.77	14.37	0.40
Iowax 2 (Waxy)	Yellow dent	1948	Ames, Iowa	14.71	14.22	0.49
III. Hi-Oil	White dent	1949	Urbana, Illinois	13.33	13.01	0.32
III. Lo-Oil	White dent	1949	Urbana, Illinois	15.38	14.71	0.67
III. Hi-Protein	White dent	1949	Urbana, Illinois	14.47	14.05	0.52
III. Lo-Protein	White dent	1949	Urbana, Illinois	15.14	14.72	0.42

<sup>a</sup>, <sup>b</sup>, <sup>c</sup>, <sup>d</sup>, <sup>e</sup>, full hysteresis loops established earlier, shown in Table II, with values for comparable samples identified by identical letters.

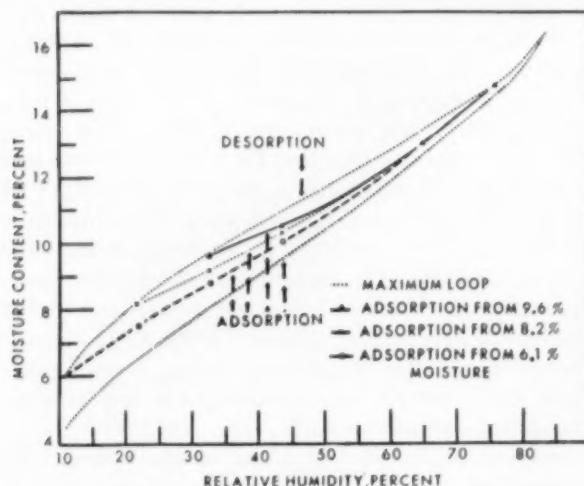


Fig. 2. Hysteresis in Pawnee wheat, 1952 crop from Illinois. Adsorption curves from varying levels of dryness superimposed over the complete hysteresis loop (maximum loop).

over a portion of the full hysteresis loop. At 56.3% humidity and higher the samples from 10 and 8% moisture reach identical equilibrium moisture contents, while the 6% sample coincides with the other two at 64.6% humidity and higher. At 75.6% humidity the adsorption curves meet and follow the desorption curve, so that no hysteresis may be exhibited above 75% humidity except in samples dried to nearly complete dryness, or, as possibly indicated above, in freshly harvested samples. From this it would appear that portions of a given lot of commercial grain differing in moisture content could be expected to reach identical moisture levels if exposed at the same temperature to a fixed humidity above 75% such as in a grain conditioner.

Rao (13) noted that hysteresis disappeared on the third cycle when "activated" polished rice was alternately wet and dried by exposure to a graded series of humidities from 0 to 100%. Pawnee wheat exposed to alternate wetting and drying over a limited series of humidities (Table V) shows the hygroscopic capacity to be essentially stable. Rao's observations on rice are apparently not applicable to wheat under the conditions of this experiment.

*Effects of Relative Humidity as Opposed to Moisture Content on Mold Growth.* An attempt was made to use the hysteresis effect to measure the relative importance of moisture content of grain and humidity of interseed air on storability of wheat and corn near the critical level for mold growth. A total of 48 ground samples of wheat and

TABLE V  
HYGROSCOPIC MOISTURE IN PAWNEE HARD RED WINTER WHEAT AT 30°C.  
OVER THE RANGE 22-76% RELATIVE HUMIDITY

SORPTION CYCLE <sup>a</sup>	RELATIVE HUMIDITY					
	22.0	32.8	43.5	56.3	64.6	75.6
%	%	%	%	%	%	%
Desorption (from 75% R.H.)						
First	8.3	9.8	11.1	12.5	13.7	
Second	8.4	9.9	11.2	12.7	13.7	
Third	8.4	10.0	11.1	12.8	13.8	
Fourth	8.4	9.9	11.1			
Fifth	8.4	9.9	11.1			
Average	8.4	9.9	11.1	12.7	13.7	
Adsorption (from 22% R.H.)						
First	...	9.4	10.5	11.9	13.2	14.8
Second	...	9.5	10.7	12.1	13.3	14.9
Third	...	9.5	10.8	12.2	13.3	14.8
Fourth	...	...	...	12.2	13.4	14.8
Fifth	...	...	...	12.1	13.1	
Average	...	9.5	10.7	12.1	13.3	14.8
Average difference	...	0.4	0.4	0.6	0.4	

<sup>a</sup> Eighteen samples cycled three times and some carried a fourth and fifth time to some levels.

corn were inoculated with xerophytic molds common to stored grain to ensure the presence of viable spores of *Aspergillus candidus*, *A. glaucus*, *A. amstelodami*, and *A. niger*. Twelve samples desorbing were exposed to an atmosphere of 72.8% and twelve to 75.6% humidity. Twelve samples adsorbing were exposed to 75.6% and twelve to 78.2% humidity at 30°C. and stored up to 6 months or until molded. Moistures were calculated from weight changes and mold activity estimated with the aid of a hand lens and a microscope.

At 72.8% humidity, samples had a range in moisture content of 14.0 to 14.4% and showed no mold growth after 168 days' storage. At 75.6% humidity, with a range in moisture of 14.6 to 14.9%, some samples molded in 69 days while others showed no mold after 168 days at the termination of the experiment, irrespective of whether they were desorbing or adsorbing moisture. Samples failing to mold included some with 14.9% moisture. At 78.2% humidity the samples had only a slightly higher range in moisture (14.7 to 15.0%), but all samples showed mold growth by the 46th day with some molds sporulating. These observations suggest that humidity may be more critical than moisture content for grain spoilage.

From the hysteresis loop in Fig. 3 it may be noted that wheat stored at 14% moisture will show an equilibrium humidity of 69 to 73% at

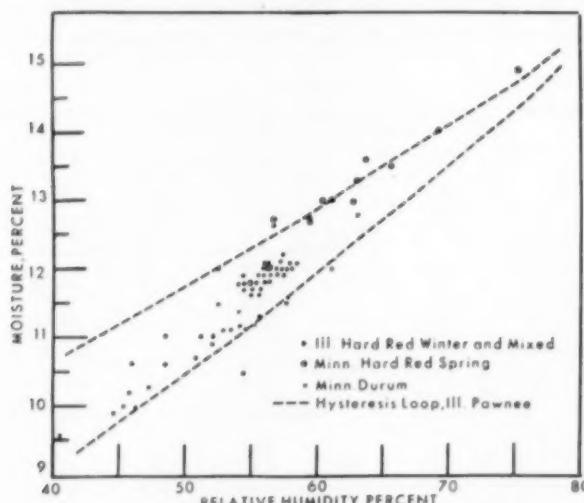


Fig. 3. Equilibrium relative humidity of interseed air in wheat as measured with the Dunmore Hygrometer, vs. moisture content as measured with the Tag-Heppenstall moisture meter, plotted over a portion of the hysteresis loop for Pawnee hard winter wheat.

30°C. This, by most accepted standards, should be safe to store for moderately extended periods. If the temperature is raised only 5° to 35°C. (Fig. 1) the equilibrium humidity is raised to 76–79% and mold growth can occur. Such temperature changes are not uncommon to stored grain as warmer summer weather approaches, particularly in localized portions of a bin.

*Relative Humidity of Air in Equilibrium with Commercial Samples of Grain as Received.* The rapid determination of moisture content and equilibrium relative humidity by means of the Tag-Heppenstall moisture meter and the Dunmore Hygrometer offered a means of obtaining data on the moisture-relative humidity relationship for a large number of grain samples in their condition as received. Results of these measurements are shown in Fig. 3 and Fig. 4 plotted over partial hysteresis loops for wheat and corn. The spring wheat samples are grouped higher in moisture along the desorption curve, whereas the winter wheat samples are grouped more centrally in the loop and at a lower moisture level. The corn samples also show two groupings.

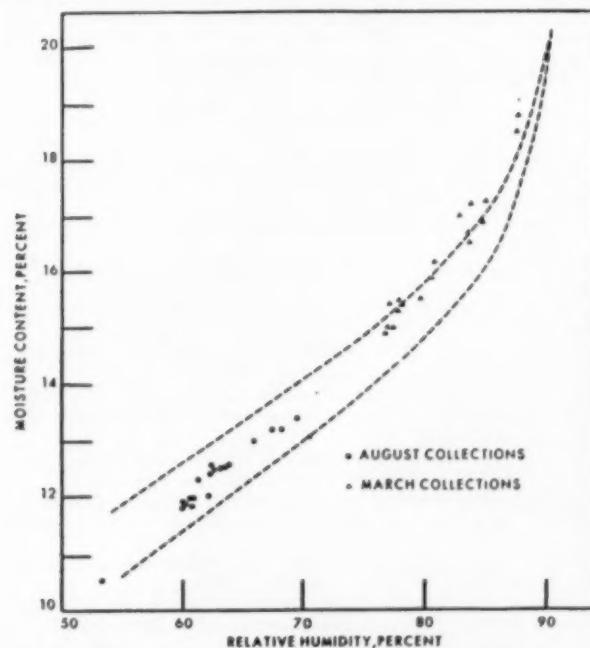


Fig. 4. Equilibrium relative humidity of interseed air of corn as measured with the Dunmore Hygrometer, vs. moisture content as measured with the Tag-Heppenstall moisture meter, plotted over a portion of a hysteresis loop for corn.

These consist of old corn collected in August 1954, which are grouped centrally within the hysteresis loop in the 12-14% moisture range, and new corn collected in March 1955, in the 15-18% moisture range, grouped around the desorption curve.

The agreement of these samples with the data from the samples for which complete isotherms were determined appears to be within the range of experimental and instrumental errors involved. It seems reasonable to assume that the samples received at higher moisture levels from late fall harvest had no opportunity to dry below the moisture content as received. The grouping of these samples along the desorption curves lends credence to this assumption for both wheat and corn. The grouping of the samples of grain from summer collections midway in the loop may indicate that they have been subjected to some cycling as they were more exposed to periods varying in humidity and temperature.

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## A RAPID METHOD FOR THE PRODUCTION OF FLOUR FOR TESTING BY FARINOGRAPHY<sup>1</sup>

JEFF SCHLESINGER<sup>2</sup>

### ABSTRACT

Flour for making farinograms was obtained by using the contact rolls of the Weston (formerly Tag-Heppenstall) moisture meter and a Roto-matic sifter. Calculations on data for 195 Southwestern hard winter wheats of the 1956 crop milled by the rapid method and by the Buhler mill indicate significant correlation coefficients, varying from 0.47 for the initial phase to 0.86 for stability.

Farinograph curves present possibilities for classifying carlots of wheat prior to unloading, provided that flour for the testing can be produced rapidly enough. A number of studies have been made in which various methods of experimentally milling wheat have been compared, but all of them involve considerable time (2, 3, 4, 5, 6, 7, 8). The purpose of this study was to devise and evaluate a more rapid method employing the contact rolls of the Weston (formerly Tag-Heppenstall) moisture meter and a Roto-matic sifter. The study involved milling 195 samples of wheat by the rapid method and compar-

<sup>1</sup> Manuscript received March 14, 1957.

<sup>2</sup> Union Equity Co-operative Exchange, Enid, Oklahoma.

ing the farinograph curves with those obtained on flours from the same wheats obtained in a Buhler mill.

### Equipment

A set of Weston moisture tester contact rolls<sup>3</sup> was re-gearred from direct to chain drive to eliminate noise and wear. The powered roll speed was 48 r.p.m. The idler or second roll revolved only when the wheat stock was pulled between the rolls by the powered roll. This produced a shearing action rather than a grinding action. Except for noise and wear, the original gear train worked satisfactorily.

The Roto-matic sifter<sup>4</sup> equipped with No. 30 wire cloth (27 meshes per lineal in.), 10XX flour silk, and a blank tray were used for bolting the stock. A 2-in. throw and 188 r.p.m. were employed.

The Buhler experimental mill was the new pneumatic model MLU-202, clothed and operated as suggested by the manufacturer.<sup>5</sup>

Two Brabender Farinographs were used, 1955 and 1956 model single-speed, 59 r.p.m., equipped with 50-g. stainless-steel bowls. The temperature was controlled by a single pressure thermostat set at 30° ± 0.1°C.

### Methods

The roll spacing of the Weston machine was adjusted to 0.40 in. using both the wheat and corn shims. A closer setting than this resulted in severe strain upon the motor and gears. A wider setting decreased the milling action and hindered the production of flour.

Untempered wheat (450 g.) was passed through the rolls five times, taking less than 2 minutes, and the ground material was sifted 3 minutes on the Roto-matic sifter. Within 5 minutes sufficient flour was obtained, at an extraction of approximately 15% for one farinograph curve using the 50-g. bowl. By keeping adequate records of the farinograph absorption required by wheats from various locations and crop years, another grinding was usually unnecessary. The ash content, averaging 0.65%, was variable, being influenced by the moisture content and the cleanliness of the sample.

A 1500-g. portion of each wheat sample was milled on the Buhler pneumatic experimental mill. It was tempered for 18 hours to 15.5% moisture and milled in an air-conditioned room at 77° ± 2°F. and 50 ± 2% relative humidity. The Buhler extractions averaged 67% with an average ash content of 0.40%.

A constant flour weight on an as-received moisture basis was used

<sup>3</sup> Weston Electrical Instrument Corp., Newark, N. J.

<sup>4</sup> General Mill and Equipment Co., Kansas City, Mo.

<sup>5</sup> Buhler Instruction Manual 6324-E.

in the two farinographs which were standardized with regard to each other. The term "initial phase" refers to the period of time from the first addition of water until the curve crossed the 500 B.U. reference line. The terms "dough development time," "stability," and "tolerance index" are defined in *Cereal Laboratory Methods* (1).

### Results

The flours milled by the rapid method and by the Buhler method produced similar farinograph curves. The statistics in Table I were derived from the data from 195 samples of 1956 crop hard winter wheat grown at 54 Southwestern locations. Farinograph absorption

TABLE I  
DATA FROM 195 SAMPLES OF 1956 CROP HARD WINTER WHEAT  
GROWN AT 54 SOUTHWESTERN LOCATIONS

N = 195	INITIAL PHASE		DOUGH DEVELOPMENT		STABILITY		TOLERANCE INDEX	
	Rapid (x)	Buhler (y)	Rapid (x)	Buhler (y)	Rapid (x)	Buhler (y)	Rapid (x)	Buhler (y)
	minutes	minutes	minutes	minutes	minutes	minutes	units	units
Range	1.5-5.0	1.5-5.0	2.5-9.0	3.3-9.0	2.5-18.0	2.8-19.5	10-80	5-85
Means	3.29	3.22	6.09	6.18	11.51	11.72	26.79	24.33
Std. dev.	0.61	0.67	0.99	0.94	3.01	3.32	13.96	13.85
Corr. coeff. ( $r_{xy}$ )	0.467**		0.700**		0.861**		0.803**	
Std. error of estimate	0.59 minutes		0.67 minutes		1.68 minutes		8.25 units	
Regression equations	$y=1.547+0.5085x$		$y=2.14+0.663x$		$y=0.797+0.949x$		$y=2.98+0.797x$	

values were not analyzed statistically, as the rapid method used untempered wheat while the Buhler method used tempered wheat. Val orimeter data were not obtained.

An examination of the means and standard deviations reveals good agreement and demonstrates the absence of systematic error of consequence.

Correlation coefficients ranged from a low of 0.467 for initial phase, the least important of the measurements taken, to 0.861 for stability. All correlations were statistically significant.

The standard error of estimate (9) measures the ability of the results of one method to predict the probable results of the other. The data indicated that the farinograph curves from flour milled by the rapid method will probably check with Buhler flour curves within 0.59 minute for the initial phase, 0.67 minute for dough development time, 1.68 minutes for stability, and 8.25 units for the tolerance index.

The regression equation ( $y = a + bx$ ) gives the information that is necessary for the prediction of the Buhler mill procedure from the data obtained by the rapid method. X was the value obtained by the rapid milling method and y by the Buhler.

The rapid milling method has practical merit for those who need to obtain quickly farinograph curves on numerous wheat samples. The method has been and is currently being employed in the author's laboratory to obtain data subsequently used by the elevator superintendents to classify, bin, and blend their wheat stocks. The resulting blends have been entirely satisfactory with regard to farinograph curve characteristics.

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## SOME EFFECTS OF ENVIRONMENT AND VARIETY ON THE AMYLOSE CONTENT OF BARLEY<sup>1</sup>

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### ABSTRACT

The amylose content of starch obtained from 30 barley samples (Compana variety) representing different environmental and cultural practices ranged from 19 to 23% on the dry basis. The amylose content of 44 different varieties varied from 15 to 24%, indicating inherent differences.

Fifteen genotypes grown from spikes containing barley kernels with shrunken endosperms were not different from other varieties in amylose content. Determinations on five isogenic pairs showed no association with the gene under study, with the possible exception of the hooded character.

The amylose content of five genetic "freaks" ranged from 11 to 26%.

Recently there has been considerable interest in finding cereals with high amylose content (3, 4), because it has been suggested that starch with high amylose content might be used for the preparation of films with characteristics like those for pure amylose described by Wolff *et al.* (7). If such starch could be obtained, it might replace a considerable amount of alpha cellulose and this, in view of the shortage of wood pulp and the excess of cereal grains, would be highly desirable.

Since acreage limitations have been established on the amount of land which can be planted to wheat, large surpluses of barley have been accumulating in the Northwest, because barley is about the only other cereal that can be successfully grown on much of this wheat land. The present investigation was undertaken to determine whether cultural treatments or environment would influence the amylose content of Compana barley starch. Along with this study a number of other barley samples of different varieties were analyzed with the hope that differences in amylose content would appear, indicating that by breeding, a variety of high amylose content could be developed.

### Materials and Methods

Thirty barley samples of the Compana variety representing different years, geographical locations, fertilizer treatments, and stage of maturity were available for study.

From nurseries grown in Bozeman in 1955, 26 varieties of commercial importance or selections of promise from the agronomic stand-

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point and 19 varieties of barley of foreign origin recently introduced into the United States were obtained.

Over a period of years, spikes that contained barley kernels with noticeably shrunken endosperms at maturity had been collected at Bozeman. These were grown in the field in 1955.

To determine if isogenic barleys would have different amylose contents, three glossy-stemmed mutants were studied; this series was supplemented with one variety, Atlas 46, and a hooded form obtained by backcrossing.

As a result of an extended period of investigation, workers at the Aberdeen Branch Station in Idaho collected barley mutant or recombination types. Five of these were analyzed.

The barley samples were pearly rather severely to remove the lemma and palea, most of the pericarp, and a considerable fraction of the germ. The remaining endosperm was ground in a Wiley mill through a 40-mesh screen and the fat extracted with 85% methanol (by volume) for 24 hours prior to dispersion in alkali. A portion of the methanol-extracted endosperm was analyzed for protein, moisture, fiber, and ash.

The nitrogen was determined by a modified Kjeldahl method (1, p. 343) and the result multiplied by the factor 6.25; the moisture by determining loss in weight after drying in a vacuum for 24 hours at 100°C.; and the fiber and ash by the standard A.O.A.C. procedures (1, pp. 13, 346).

The values given for starch content were obtained by subtracting the sum of the moisture, ash, fiber, and protein from the total. These samples were stored over the winter in a dry room; this resulted in their moisture content being nearly constant. The sum of moisture, ash, and fiber averaged 10.0% with a maximum error of  $\pm 0.2\%$ . Therefore, this figure was used to calculate starch content of samples. These figures do not give absolute values for starch; but since a total error of 1% would make an error of only 0.25% in the amylose determination, which is well within the experimental error of the method, the procedure was considered adequate.

To determine if the protein present in the barley flour would affect iodine absorption and thus give misleading results, starch was isolated from eight samples of barley flour which varied in protein content from 7.9 to 18.4%. These all gave the same values for amylose regardless of whether the flour or starch were used. However, there is some indication that the bran does contain a factor which changes the values obtained for amylose. Since this was removed by severe pearlying the results were not influenced. The authors would recommend separation

of the starch before analysis in any future work, since it would conserve time and eliminate other possible errors.

The amylose content was determined by a potentiometric titration with iodine using the method described by Bates, French, and Rundle (2) as modified by Lansky *et al.* (5); the calculations of percent amylose were based on the fact that 1 g. of barley amylose combines with 0.215 g. of iodine (6).

TABLE I  
INFLUENCE OF CULTURAL PRACTICES AND ENVIRONMENT ON PERCENT AMYLOSE IN  
COMPAGN BARLEY STARCH

	PROTEIN	STARCH	AMYLOSE IN STARCH
	%	%	%
<b>YEAR HARVESTED (Bozeman, irrigated)</b>			
1950	14.1	75.9	21
1951	16.1	73.9	21
1952	16.0	74.0	21
1953	18.6	71.4	21
1954	10.1	79.9	21
1955	10.6	79.4	21
<b>FERTILIZER TREATMENT (Kalispell, dryland, 1954)</b>			
Type	Analysis	lb./acre	
Ammonium nitrate	33.5-0-0	100	13.8
Ammonium sulfate	21-0-0	120	13.6
Ammonium phosphate-sulfate mix	16-20-0	60	10.2
Ammonium phosphate	11-48-0	300	7.3
Superphosphate	0-43-0	325	8.9
<b>PLANTING DATE (Bozeman, irrigated, 1954)</b>			
May 4		7.2	82.8
May 18		7.2	82.8
June 3		7.9	82.1
June 17		7.9	82.1
<b>LOCATIONS, 1955</b>			
Dryland			
Creston		7.9	82.1
Sidney		11.1	78.9
Huntley		13.3	76.7
Havre		10.3	79.7
Springhill		12.2	77.8
Irrigated			
Creston		7.9	82.1
Sidney		9.6	80.4
Stevensville		8.3	81.7
Missoula		11.1	78.9
Ronan		12.8	77.2
Bozeman		10.6	79.4
<b>GROWTH STAGE WHEN HARVESTED</b> (Bozeman, irrigated, 1955)			
Late milk		13.3	76.7
Soft dough		10.3	79.7
Hard dough		10.3	79.7
Ripe		9.3	80.7

### Results and Discussion

*Effect of Environment and Cultural Practices on Amylose Content of Compana Barley Starch.* The effects of fertilizer treatment, date of planting, moisture level, stage of maturity when harvested, geographical location, and combined effect of year grown and length of time in storage on the amylose content of Compana barley starch are shown in Table I.

These variables had little or no measurable effect on the amylose content of Compana barley starch.

Since environmental influence and cultural practices were found to be insignificant, it was possible to examine samples from any available source with some assurance that any marked differences noted would be genetic differences due to variety.

*Percent Amylose in Barley Starch from Barleys of Commercial Importance in the United States and Canada.* The amylose contents of

TABLE II  
PERCENT AMYLOSE IN BARLEY STARCH FROM BARLEYS OF COMMERCIAL IMPORTANCE IN THE UNITED STATES AND CANADA, AND FROM CERTAIN SELECTIONS

VARIETY OF SELECTION	C.I. OR MONT. SELECTION NO.	PROTEIN	STARCH	AMYLOSE IN STARCH
				%
Two-row varieties and selections				
Dekap	3351	9.0	81.0	24
Otis	7557	10.0	80.0	22
Two-row	7837	9.2	80.8	22
Compana	5438	10.6	79.4	21
Spartan	5027	14.6	75.4	21
Compana × Morgenrot	49-527-24	11.7	78.3	19
Moravian	7559	10.4	79.6	19
Sanalta	6087	10.5	79.5	18
Glacier × Compana	47-7405-V	9.4	80.6	16
Six-row varieties and selections				
C.I. 5461 bulk, F <sub>20</sub>	9183	9.5	80.5	24
Lico III	9181	10.5	79.5	23
Barbless	5105	18.4	71.6	23
Atlas 46	7323	8.9	81.1	23
Husky	9537	9.5	80.5	23
Titan	7055	10.4	79.6	23
Velvone 11	7088	9.4	80.6	22
Glacier × Titan	50-5639-12	11.7	78.3	21
Trebi	936	9.0	81.0	20
Harlan	7008	9.6	80.4	20
Traill	9538	9.4	80.6	20
Prosser No. 6	10087	9.4	80.6	19
Bonneville	7248	8.8	81.2	18
Tammi	8345	9.3	80.7	17
Hiland	9530	10.1	79.9	17
Custer	8053	14.2	75.8	15
Mars	7015	11.7	78.3	14

barley starches from a number of barley varieties of commercial importance and from selections of promise from the agronomic standpoint are recorded in Table II.

There are inherent differences in the barley varieties studied, but there was no appreciable difference between the 2-row and the 6-row types of barley. However, none of the varieties was abnormally high in amylose content.

*Percent Amylose in Barley Starches of Foreign Origin.* The amylose contents of a number of varieties of foreign origin which were grown in Bozeman during the year 1955 are given in Table III.

TABLE III  
PERCENT AMYLOSE IN BARLEY STARCHES FROM BARLEYS OF FOREIGN ORIGIN

PLANT INTRODUCTION NUMBER	COUNTRY OF ORIGIN	PROTEIN	STARCH	AMYLOSE IN STARCH
				%
221377	Yugoslavia	13.4	76.6	19
221322		13.3	76.7	17
221309		13.7	76.3	16
221304		15.0	75.0	16
221327		14.6	75.4	15
212847	Afghanistan	16.5	73.5	15
219860		16.3	73.7	15
221422		12.2	77.8	15
212850		12.4	77.6	14
211598		13.1	76.9	14
212845		16.8	73.2	13
117532	Pakistan	15.1	74.9	18
220069	Pakistan	15.0	75.0	13
220853	Sweden	19.0	71.0	17
215708	Peru	17.0	73.0	16
221072	Germany	16.7	73.3	15
216035	India	16.4	73.6	20
214326	India	15.0	75.0	15
219757	South Africa	13.6	76.4	18

There were no consistent differences in the percent amylose of the starch of barley varieties originating in the different countries. Furthermore, no varieties of high amylose content were observed.

*Percent Amylose in Starches from Barley Grown in 1955 from Selected Shrunken Endosperm Spikes.* For some years spikes that contained, for no apparent reason, barley kernels with noticeably shrunken endosperms at maturity had been collected at Bozeman. On the assumption that these varieties might show some inherent differences in starch type, some of them were selected and grown in the field at Bozeman in 1955. These samples did not produce any inherent shrunken-endosperm types. Their analyses, given in Table IV, show a rather wide

TABLE IV  
PERCENT AMYLOSE IN STARCHES FROM BARLEY GROWN IN 1955 FROM  
SELECTED SHRUNKEN ENDOSPERM SPIKES

1955 BOERMAN: Row Number	PROTEIN	STARCH	AMYLOSE IN
			STARCH
7158	11.4	78.6	20
7157	12.7	77.3	20
7162	12.1	77.9	18
7163	11.2	78.8	18
7166	11.6	78.4	18
7159	12.3	77.7	18
7154	17.8	72.2	17
7165	13.4	76.6	17
7164	12.3	77.7	17
7169	12.6	77.4	17
7168	10.1	79.9	16
7167	11.7	78.3	16
7160	13.3	76.7	15
7155	17.6	72.4	15
7152	18.2	71.8	13

range in amylose content, but observed variations were similar to those found for different varieties and selections (Table II).

The amylose contents of the starch from glossy (nonwaxy) stemmed mutants of three varieties of barley are compared in Table V with the normal variety from which the mutant arose. The differences were small and are probably not significant except in the case of Mars barley. Atlas 46 was compared with a hooded form obtained by back-crossing. The difference of 7% (Table V) in the amylose content of the starch from these two strains indicates a real genetic difference.

*Percent Amylose in Starches of Barley "Freaks."* Over a period of years, workers at the Aberdeen Branch Station in Idaho have collected barley mutant or recombination types from the extensive barley pro-

TABLE V  
PERCENT AMYLOSE IN STARCHES FROM ISOGENIC BARLEY PAIRS

VARIETY	ISOGENIC TYPE	PROTEIN	STARCH	AMYLOSE IN
				STARCH
Barbless		18.4	71.6	23
Barbless	Glossy mutant	10.1	79.9	23
Mars		11.7	78.3	15
Mars	Glossy mutant	8.9	81.1	17
Compana		10.6	79.4	21
Compana	Glossy mutant	14.5	75.5	20
Compana	Seedling stripe, glossy mutant	9.4	80.6	20
Atlas 46		8.9	81.1	23
Atlas	Hooded	10.5	79.5	16

TABLE VI  
PERCENT AMYLOSE IN STARCHES OF BARLEY "FREAKS"

1955 BOZEMAN ROW NUMBER	ABERDEEN No.	DESCRIPTION	PROTEIN	STARCH	AMYLOSE IN THE STARCH
					%
7219	1223	Triple beard	11.5	78.5	26
7223	1236	Slit awn	11.7	78.3	23
7220	1228	Triple beard	7.7	82.3	19
7217	1221	Um, long-awned laterals	16.0	74.0	19
7218	1222	Triple beard, two collars	15.1	74.9	11

gram there. Five of these "freaks" were grown at Bozeman and the results of their analysis are given in Table VI.

The percent amylose ranged from 11 to 26% and represents the greatest differences observed in this study. It would appear that this group of material, which is represented by several hundred lines, might be the most fertile field for further search for genes conditioning amylose content. Intercrossing of high or low amylose content lines and examining segregating progenies for transgressive segregation would seem to offer a possibility for the isolation of lines higher or lower in amylose content than those observed in this study.

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## CHANGES IN ROUGH RICE OF DIFFERENT MOISTURE CONTENT DURING STORAGE AT CONTROLLED TEMPERATURES<sup>1</sup>

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### ABSTRACT

Caloro rough rice samples containing 11.2, 13.8, 15.3, and 16.5% moisture were stored in loosely covered cans containing humidifying solutions for 7 months at 70°F. (21°C.) and 90°F. (32°C.) and tested monthly for moisture, odor, viability, milling yield, free acidity, monocarboxyl compounds, total and reducing sugars, and counts of molds, yeasts, actinomycetes, and aerobic and anaerobic bacteria.

Changes in characteristics occurred more quickly and more extensively as moisture content or temperature increased, but milling yields remained unchanged until quality had seriously deteriorated. Only the two low-moisture rices at 70°F. (21°C.) remained free from sourness. Oxidative changes were not detected.

Populations of actinomycetes, bacteria, and yeasts decreased under all storage conditions, though yeast reductions were small at 70°F. (21°C.) for rice of 11.2 and 13.8% moisture. Molds increased in the two high-moisture rices, extensively at 16.5% moisture at both temperatures. Nonreducing sugars decreased during storage, whereas reducing sugars and free acidity increased. Acid increases were logarithmic with time, and the rates increased approximately exponentially with moisture content of the rice.

Percentages of germination and nonreducing sugars and log of free acidity, representing the three most sensitive characteristics, were closely related and had high coefficients of linear correlation. Changes in these storage effects also showed trends in agreement with mold growth, a potential cause.

Present international rice marketing conditions have required the prolonged storage in the United States of much larger than usual amounts of rough rice. The practical problems of bulk storage in farm or commercial bins are being extensively studied, and a recent summary of results and recommendations is now available (3).

These practical problems have emphasized the need for systematic study of individual storage factors under controlled conditions of temperature and moisture, so that the effect of these variables on the grain properties can be separately evaluated. Little has been done in this direction for rice, as is clearly evident in the excellent survey, *Storage of Cereal Grains and Their Products*, edited by Anderson and Alcock for the American Association of Cereal Chemists (1).

Several storage factors have been examined. Christensen (6) has re-

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cently summarized effects of molds on deterioration of stored grain. Teunisson (21) showed that molds, anaerobic organisms, and aerobic bacteria were predominant in air-dried rough rice harvested by combine, and that yeasts became predominant while viability decreased in moist rice under hermetic storage. The work of Del Prado and Christensen (8) demonstrated that fungi on rough rice were in general similar to those of other grains. The molds on rice in hermetic storage increased with increasing moisture content above 15% at temperatures of 63° to 75°F. (17° to 24°C.). In samples with 15.3 to 18.9% moisture, aerated 18 days at room temperature, the only mold found was *Aspergillus glaucus*. The decrease in viability as *A. glaucus* increased suggested an effect of the fungus on the germ.

In the present investigation, changes have been measured in several characteristics of rough rice of four different moisture contents between 11.2 and 16.8% during 7 months of storage at 70° and 90°F. (21° and 32°C.). Comparable studies made concurrently on the same rice stored in 1000-bushel bins (14) allowed comparisons between laboratory and bulk storage. The more drastic conditions used in some of the laboratory tests indicated what could be expected in bin storage over prolonged periods of unfavorable atmospheric conditions. Moreover, the different closely controlled laboratory storage conditions permitted partial evaluation of the separate effects of changes in moisture content and of increase of temperature on the several characteristics of the rice during prolonged storage.

### Materials and Methods

Caloro rice of the 1953 California harvest was taken directly from the dryers as a portion of material prepared for bin-scale storage tests. It was delivered to the laboratory at 11.2, 15.3, and 15.7% moisture (Tag-Heppenstall moisture meter). The 15.7% moisture sample was allowed to dry in the sack at room temperature for 4 days to yield paddy with 13.8% moisture. A separate supply of undried Caloro with 16.5% moisture (Tag-Heppenstall) was also obtained. Storage was begun the second day after receipt or drying.

The rice was filled into square 5-gal. tins to within 4 in. from the top. Containers of humidifying solutions were placed on the rice and the cans were loosely covered. Rice from all four moisture levels was stored in constant-temperature rooms at 70° and 90°F. (21° and 32°C.).

Humidifying solutions chosen to hold the rices at about their original moistures consisted of saturated solutions plus excess salt (Table

TABLE I  
SATURATED SALT SOLUTIONS FOR HUMIDITY CONTROL

ORIGINAL MOISTURE IN RICE	70°F.		90°F.	
	Salt	Relative Humidity	Salt	Relative Humidity
%	%	%	%	%
11.2	Magnesium nitrate	54	Sodium nitrite	63
13.8	Sodium nitrite	65	Sodium acetate	70
15.3	Potassium chloride	84	Potassium chromate	86
16.5	Barium chloride	89	Barium chloride	90

I). They were contained in the inner of two nested crystallizing dishes to provide a large surface and to prevent contamination of the paddy.

Two samples were taken from each can of rice at monthly intervals. A first portion (about 150 g.) for microbiological examination was taken with a sterile bottle from just below the surface of the rice. A second sample of about 1,300 g. was taken by grab sampling throughout the can. A portion of this was reserved for chemical analyses and the remainder was used for milling tests. The analytical sample was freed from trash, weed seed, and dehulled kernels so that analytical data would be on the consistent basis of clean paddy rice.

Moistures were determined with the Tag-Heppenstall (Tag) moisture meter (7), monocarbonyl compounds by the previously described Pool and Klose procedure (11), and acidity of the hexane-extracted oil by the modified Ames and Licata titration (13). Total and reducing sugars were determined as glucose by A.O.A.C. methods (4), and non-reducing sugars were calculated by difference. Milling tests for total and head rice yields were made by the official U.S.D.A. procedure (23).

Viability was tested by a modified paper-towel method of the U. S. Production and Marketing Administration (22). Two 100-seed lots were incubated at 25°C. (77°F.) for 8 days. Average percentage germination of the two lots was used as a measure of viability.

For microbiological examination 50 g. of rice were weighed into a sterile Petri dish, then transferred to a sterile blender jar containing 450 ml. of sterile water. The rice was soaked 10 minutes, then subjected to high-speed blending for 1 minute. After settling 1 minute, serial dilutions were made for microbiological counts. Poured plates used were as follows:

1. For mold and yeast counts: Difco malt agar.
2. For aerobic bacteria counts: Difco Tryptone glucose extract agar.
3. For anaerobic bacteria counts: Difco brewer anaerobic agar and plain agar in combination with a special Petri dish and

glass plate as described by Andersen (2), with the inoculated anaerobic agar below the plate and the plain agar poured over the plate.

4. For actinomycetes count: Conn's soil extract agar as described by Henrici (19).

All poured plates were incubated 48 to 72 hours at 26° to 27°C. (79° to 80.5°F.).

### Results and Discussion

*Moisture.* Moisture contents remained fairly constant (Table II), except during later stages of storage of high-moisture grain. The in-

TABLE II  
MOISTURE CONTENTS OF STORED ROUGH RICE  
(Tag-Heppenstall)

STORAGE TIME days	70°F.				90°F.			
	A	B	C	D	A	B	C	D
0	11.2	13.8	15.3	16.5	11.2	13.8	15.3	16.5
33	11.1	13.6	15.3	16.5	11.1	13.4	15.3	16.5
69	11.1	13.9	15.5	16.8	11.1	13.6	15.5	16.7
90	11.2	14.0	15.5	16.7	11.2	13.6	15.5	16.9
125	11.1	13.9	—	16.9	11.4	13.5	15.6	17.0
153	11.3	14.0	15.9	17.0	11.4	13.4	15.8	17.5
185	11.1	13.2	15.7	17.0	11.6	13.6	15.7	18.9
215	11.3	13.7	—	17.2	11.6	—	15.9	19.7
Av.	11.2	13.8	15.5	16.8	11.3	13.7	15.7	16.7 <sup>a</sup>

<sup>a</sup> Average of first five values.

creases in these cases accompanied vigorous growth of molds, and the resultant moisture equilibria apparently were no longer those for rice alone. Similar effects have previously been reported (18) for wheat. The average moisture contents of the several lots of rice are used to designate them throughout the tabulated results to provide ready intercomparisons among the data.

*Odors.* Sourness developed in the two rices of highest moisture in 2 months at both 70° and 90°F. (21° and 32°C.) and after 3 months in all the rice at 90°F. (32°C.) The two low-moisture rices at 70°F. (21°C.) remained fresh over the 7-month period.

*Milling Yields.* Total and head-rice yields showed no significant changes except in the high-moisture rice held at 90°F. (32°C.). This sample showed some loss in head-rice yield after extensive quality deterioration had occurred. Milling yields were much less sensitive to storage conditions than were other quality characteristics.

*Oxidative Changes in Lipids.* Monocarbonyl values (indicative of oxidation of the oil) of the original rice were 0.056 to 0.078 micromole

per g. of moist rice. After 7 months the rice stored at 90°F. (32°C.) had values of 0.042 to 0.050, and the rice stored at 70°F. (21°C.), 0.042 to 0.060 micromole per g. The slight decreases in already low values are probably not significant. Changes in the grain, as might be expected, are not of an oxidative nature.

**Sugars.** Sugar contents (Table III) showed little change at either temperature in rice with less than 14% moisture. Rice of over 16%

TABLE III  
REDUCING AND NONREDUCING SUGARS IN STORED ROUGH RICE

STORAGE TIME days	70°F.				90°F.			
	Average Moisture, Percent				Average Moisture, Percent			
	11.2	13.8	15.5	16.8	11.3	13.7	15.7	16.7
Reducing sugars								
0	0.09	0.10	0.11	0.12	0.09	0.10	0.11	0.12
33	.06	.07	.08	.08	.06	.08	.08	.11
69	.07	.08	.10	.15	.07	.09	.09	.16
90	.08	.07	.11	.15	.06	.10	.12	.20
125	.07	.10	.12	.17	.08	.11	.14	.21
153	.08	.10	.10	.14	.08	.09	.17	.21
185	.07	.08	.11	.19	.08	.08	.18	.18
215	0.06	0.08	0.11	0.16	0.08	0.09	0.19	0.16
Nonreducing sugars								
0	0.78	0.71	0.76	0.81	0.78	0.71	0.76	0.81
33	.85	.79	.80	.78	.88	.75	.76	.71
69	.77	.74	.69	.58	.75	.72	.67	.47
90	.91	.74	.80	.65	.85	.82	.72	.45
125	.94	.85	.81	.66	.98	.82	.68	.28
153	.82	.75	.67	.56	.80	.74	.42	.14
185	.78	.72	.66	.44	.79	.72	.28	.05
215	0.75	0.72	0.54	0.46	0.77	0.74	0.27	0.04

moisture at 70°F. (21°C.) and over 15% moisture at 90°F. (32°C.) underwent appreciable to strong loss of nonreducing sugars together with some increase in reducing sugars. At 90°F. (32°C.) and 16.5% moisture the rice lost almost all its nonreducing sugars, and finally some of the reducing sugars.

**Microbiological Changes.** Microbial population of the rice entering storage consisted of aerobic bacteria, actinomycetes, and anaerobic organisms in millions per g., yeasts in 0.5 to 5 millions per g., and molds in 10 to 50 thousand per g. The counts of aerobic bacteria, anaerobes, and molds are of the same order found by others (8, 21) on rice. Actinomycetes and yeast counts were much higher than those reported by Teunisson (21). Tables IV and V show the changes in numbers of yeasts and molds during storage, and the initial and final values for other microorganisms. The outstanding feature is the large increase in

TABLE IV  
YEASTS AND MOLDS IN STORED ROUGH RICE  
(Thousands per gram)

STORAGE TIME	70°F.				90°F.			
	Average Moisture, Percent				Average Moisture, Percent			
days	11.2	13.8	15.5	16.8	11.3	13.7	15.7	16.7
Yeast								
0	5000	500	3000	610	5000	500	3000	610
33	2000	300	3000	200	3000	300	600	<0.1
69	2000	500	100	60	100	120	0.1	<0.1
90	400	1080	2600	60	14	2100	60	0.1
125	500	880	13	0.1	500	12	0.1	0.1
153	120	55	80	4	10	1	...	...
185	400	12	55	<0.1	10	<0.1	<0.01	<0.01
215	2400	450	150	<0.1	100	70	<0.01	<0.01
Molds								
0	21	20	50	10	21	20	50	10
33	6	150	3	1,000	4	40	32	8,000
69	20	4	47	10,000	10	2	2000	39,000
90	10	10	130	9,600	2	0.1	3300	25,000
125	0.1	200	850	5,600	0.1	0.1	3900	7,200
153	0.9	1	790	5,300	0.1	3	5800	16,500
185	0.5	10	500	8,400	3	2	5000	33,200
215	0.2	0.5	650	10,000	<0.01	1	6000	89,000

mold counts and the practical disappearance of yeasts in the higher-moisture rices, particularly at 90°F. (32°C.). Other microorganisms tended to decrease under all test conditions, more rapidly as temperature and moisture were increased. At 11.2 and 13.8% moisture there was a general decrease of all organisms. Critical moisture content for mold growth on rice at these temperatures appeared to be 14 to 15%. No identifications of types or species were made in this study.

TABLE V  
ACTINOMYCETES AND BACTERIA IN STORED ROUGH RICE  
(Millions per gram)

STORAGE TIME	70°F.				90°F.			
	Average Moisture, Percent				Average Moisture, Percent			
days	11.2	13.8	15.5	16.8	11.3	13.7	15.7	16.7
Actinomycetes								
0	88	64	21	41	88	64	21	41
215	28	23	10	1	6	3	0.2	<0.001
Aerobic bacteria								
0	100	124	99	30	100	124	99	30
215	52	29	15	0.8	19	8	0.3	<0.001
Anaerobic bacteria								
0	68	27	46	12	68	27	46	12
215	3	0.3	0.05	0.04	0.02	0.01	<0.001	<0.001

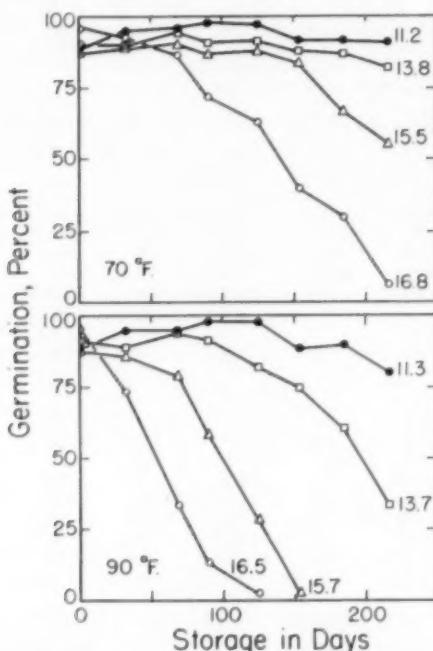


Fig. 1. Changes in viability of stored rough rice of various moisture contents (shown at end of graphs).

**Viability.** Viability is one of the two measured characteristics most sensitive to storage conditions. Figure 1 shows how the fall in percent germination became more rapid as moisture content increased, and how an increase in temperature strongly affected rice with 14% or more of moisture. The small viability increases of dry rice for the first 3 to 4 months indicate an after-ripening or post-harvest maturation. The concurrent initial opposition of this maturation tendency to those factors causing decreased viability may account for the delayed appearance of rapid viability losses in samples of intermediate moisture content.

**Acidity.** Free acidity in the oil is also very sensitive to storage conditions. Table VI illustrates how development of free acidity increased with a rise in temperature and moisture.

Increases of acidity appear due to lipolytic rather than oxidative reactions, for there was no concurrent increase in oxidation products such as monocarbonyl compounds, in contrast to the action found in parboiled rice (11) in which oxidation and acidity increased at the same time.

TABLE VI  
FREE ACIDITY IN OIL OF STORED ROUGH RICE  
(Milli-equivs. per gram oil)

Storage Time days	70°F.				90°F.			
	Average Moisture, Percent				Average Moisture, Percent			
	11.2	13.0	15.5	16.8	11.3	13.7	15.7	16.7
0	0.076	0.102	0.095	0.078	0.076	0.102	0.095	0.078
33	.076	.077	.082	.066	.083	.097	.089	.032
69	.078	.101	.102	.082	.081	.109	.107	.223
90	.076	.091	.102	.087	.087	.113	.121	.318
125	.088	(.037)*	.090	.118	.102	.135	.161	.583
153	.075	.116	.102	.124	.099	.137	.157	.598
185	.086	.114	.113	.157	.110	.154	.218	.857
215	0.087	0.123	0.114	0.169	0.117	0.159	0.228	1.25

\* Obviously in error; not used in calculations.

Acid content of the oil increased logarithmically with time for each of the conditions studied. Plots of log of free acid against time are linear, with regression coefficients of 2.75 to 55.08, all  $\times 10^{-4}$ .

A single exception is that the values for 16.5% moisture rice at 90°F. (32°C.) after 153 days fall on a separate rate line from earlier values. Data are insufficient to evaluate the significance of the fact that this occurs after viability has disappeared, mold growth has become heavy, and moisture equilibrium values have appreciably increased.

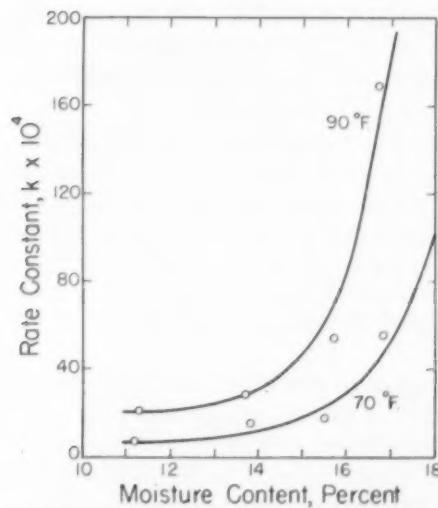


Fig. 2. Relation between rates of acid increase and moisture content of rough rice. Points represent calculated rates under individual storage conditions. Solid curves are traces of derived exponential equations.

That the rate of acid increase in rough rice follows an autocatalytic course for each set of environmental conditions can be shown by converting amounts of free acid (calculated as oleic) to percentages of the oil. Linear relations result from plotting  $\log (\% \text{ free acid}/\% \text{ unhydrolyzed oil})$  against time (10), in agreement with an autocatalytic equation.

*Interrelationships of Characteristics.* The relation of rate constants for acidity increases (slopes of the graphs for  $\log$  acidity against time) to moisture content of the rice is shown by the points in Fig. 2 at both test temperatures. Above 14.5 to 15% these rates rise rapidly with moisture increase, though at 70°F. (21°C.) the change is more abrupt than at 90°F. (32°C.).

The approximation of this relation to an exponential increase is also shown in Fig. 2 by the relation of the points to the solid curves. These are exponential curves, based on the experimental data, of the form:  $k = ae^{bx} + c$ , in which  $k$  is the rate of acidity increase corresponding to moisture percentage,  $x$ , in the rice,  $e$  is the base of natural logarithms, and  $a$ ,  $b$ , and  $c$  are numerical constants. Plots of  $\log (k - c)$  against  $x$  are linear with slope  $b \log e$ , and intercept  $a$ . This is the same type of curve as that found by Bailey (5) for changes in respiration rates of rice and other grains with variation in moisture content at 77°F. (25°C.).

Later work of Milner and Geddes (16) has related the changing respiration rates for grains in general to changes in activity of the microflora present. Table IV of the present study shows that mold activity is the one most in accord with this behavior.

Sorger-Domenigg and co-workers have pointed out the limitations of mold counts as indices of deterioration of wheat during storage under conditions causing appreciable reductions in mold populations (20). Such a limitation is accepted for rice, and only those present storage series showing increases in mold population have been used for comparison of mold counts with changes in other grain characteristics. These are the two high-moisture series at 70° and 90°F. (21° and 32°C.).

A moderately good relationship is found between increases in  $\log$  of mold population and decreases in viability or in nonreducing sugars, and a somewhat lower agreement is shown between  $\log$  of mold populations and  $\log$  of free acidity. Scattergrams for these relations show, however, that they are not linear and that simple correlation coefficients are not applicable.

*Interrelationships among the three sensitive characteristics of vi-*

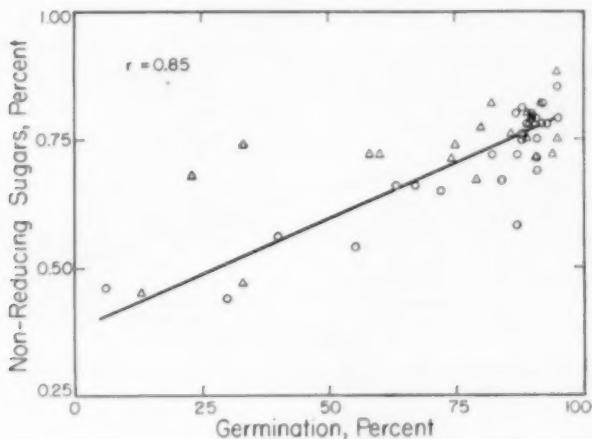


Fig. 3. Relation between percentage germination and percentage of nonreducing sugars; circles indicate storage at 70°F. (21°C.); triangles, 90°F. (32°C.).

ability, free acidity, and nonreducing sugars are, however, very good. Scattergrams presented in Figs. 3, 4, and 5 illustrate these relations. The log of free acidity is proportional to the other two characteristics. Zeleny and Coleman, who worked with corn (24, 25), and Sorger-Domenigg *et al.*, who worked with wheat (20), found the acidity correlated with the log of viability. If log of acidity is correlated with viability for Zeleny and Coleman's data (25), the coefficient is  $-0.88$  instead of their value of  $-0.85$ . Moreover, the present data for rice

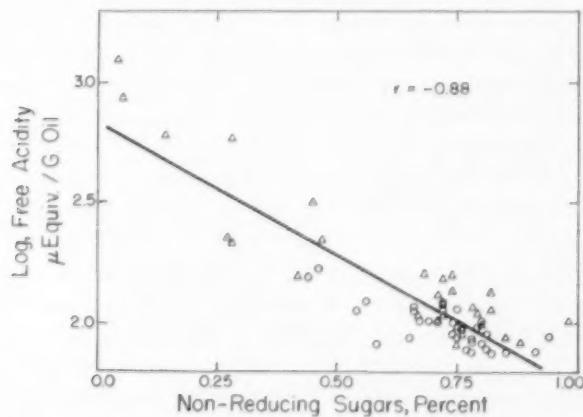


Fig. 4. Relation between free acidity and percentage of nonreducing sugars; circles indicate storage at 70°F. (21°C.); triangles, 90°F. (32°C.).

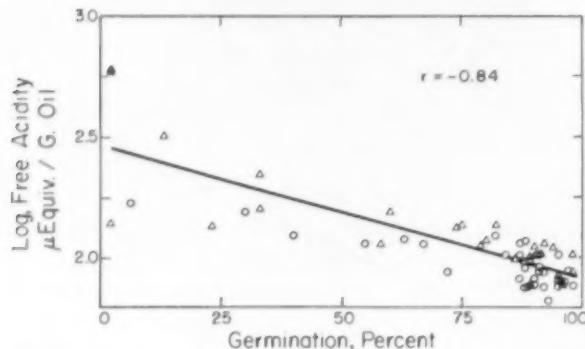


Fig. 5. Relation between free acidity and percentage germination; circles indicate storage at 70°F. (21°C.); triangles, 90°F. (32°C.).

give a coefficient of  $-0.82$  with the procedure of Sorger-Domenigg *et al.* (20) as against the present  $-0.84$ . The differences within each of the two pairs of correlation coefficients are not statistically significant. The agreement among these several investigators working with different cereals emphasizes the generality of interrelated changes in quality characteristics of cereals during deterioration.

### Conclusions

Rough rice reacts generally to storage conditions in a manner expectedly similar to that of other grains, and high degrees of correlation are found among the changes occurring in the most sensitive characteristics of the rice. Within this generality it has been found that acidity development with time follows an autocatalytic course. In addition, the change in rate of this development with change in moisture content approximates an exponential relation. Regularity is thus shown among changes resulting from storage, and between the results and the conditions of storage.

No comparable degree of regularity has developed between the results and a causative agent. The data are in agreement with the reports by Geddes, Christensen, and co-workers (6, 12, 15, 17, 18, 20) on the importance of molds in grain deterioration, and with the suggestion (9) that mold lipase may be responsible for the development of fat acidity. Although the challenging problem of attempting to relate lipase activity to the changes found is not presently contemplated, further investigations are being made to learn whether the reported regularities will be confirmed and extended by tests on rough rice from other crop years and under variation in storage conditions.

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## A RAPID TURBIDIMETRIC METHOD FOR DETERMINATION OF ZEIN<sup>1</sup>

E. M. CRAINE, CAROL A. JONES, AND JOYCE A. BOUNDY

### ABSTRACT

Zein dissolved in an ethanol-water system is simply and rapidly determined by measurement of the turbidity which occurs when a solution of 1% sodium chloride is added to the zein solution. In the concentration range of 100 to 1000  $\mu\text{g}$ . the turbidity was measured with a spectrophotometer at a wave length of 590  $\text{m}\mu$ . At concentrations less than 50  $\mu\text{g}$ . a nephelometer was used to obtain greater sensitivity.

The use of turbidimetric methods in analysis has been reviewed by Wells (7). Such methods have found application in the determination of proteins (6). The rapid addition of water to solutions of zein in ethanol-water systems produces a precipitation of the protein. Under certain conditions the action results in a stable suspension of the protein particles. The reproducibility of the suspensions is the basis of a new method reported here for determination of prolamines. Conditions are described whereby this method can be used for the assay of zein. The method is particularly well adapted for use in chromatography and zone electrophoresis.

<sup>1</sup> Manuscript received April 15, 1957. Contribution from the Northern Utilization Research and Development Division, Peoria, Illinois, one of the Divisions of the Agricultural Research Service, U. S. Department of Agriculture.

### Experimental Procedure

For standardization of the technique, a sample of commercial zein<sup>2</sup> was used. The protein contained 14.7% nitrogen (dry basis) and 6.9% moisture, but for simplicity the weights of zein described are not corrected for the moisture content.

*Turbidimetric Assay.* The basic procedure was the rapid addition of an aliquot of protein precipitant to a suitable aliquot of zein dissolved in an ethanol-water system. The use of water as a precipitant produced a very stable dispersion, even at relatively high concentrations, but the reproducibility was poor. When 1% saline (1 g. sodium chloride per 100 ml.) was used, the stability was not as good but the reproducibility was improved. The procedure which is recommended and which was used in the range 100 to 1000  $\mu\text{g}$ . was to place a 2-ml. aliquot of zein dissolved in 70% ethanol (vol/vol.) in a standardized 18 by 150-mm. tube. Six milliliters of 1% saline were "blown" into the sample from a 10-ml. serological pipet. The blowing gave sufficient mixing and further agitation was avoided. Small bubbles which formed on the tube surface were removed by carefully rotating the tubes at an angle. After 60 minutes the absorbance resulting from the turbidity was measured at 590  $\text{m}\mu$  by means of a Bausch & Lomb Spectronic 20 colorimeter.

*Nephelometric Assay.* To obtain greater sensitivity at lower ranges (0-40  $\mu\text{g}.$ ), the scatter of light by the suspended particles was measured with a Coleman Photo-Nephelometer Model 7 set at maximum sensitivity. The assay was performed in a manner similar to that described for the turbidimetric assay. In this concentration range the best results were obtained by placing a 1-ml. aliquot of zein dissolved in 70% ethanol (vol/vol.) in a standardized 18 by 150-mm. tube. Six milliliters of 1% saline were "blown" into the sample and after 10 minutes the scatter of light was measured. Values reported are units from a galvanometer deflection scale which had a range from 0 to 12.

*Dialysis of Zein.* Twenty-milliliter aliquots of a zein solution (70% ethanol) containing 10.0 mg. of zein per ml. were placed in sections of cellophane dialysis tubing. The individual aliquots were dialyzed against 1 liter of either 50, 70, or 95% ethanol for 24 hours. The amount of nitrogen present before dialysis was determined on three aliquots in dialysis tubing. Nitrogen was determined by a macro-Kjeldahl technique after the ethanol was removed by heating the flasks on a steam bath.

<sup>2</sup> Unmodified zein (Batch HV-91) of Corn Products Refining Company, Argo, Illinois. Mention of firm names or trade products does not imply that they are endorsed or recommended by the U. S. Department of Agriculture.

### Results

*Turbidimetric Assay.* Figure 1 shows the absorbance observed at various time intervals and zein concentrations when 1% saline was

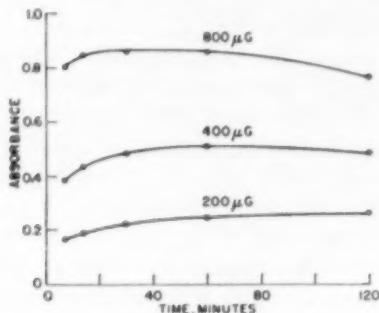


Fig. 1. Absorbance resulting from mixing 2 ml. of zein in 70% ethanol with 6 ml. of 1% saline at time intervals in minutes after the mixing. Each curve represents a tube containing either 200, 400, or 800  $\mu\text{g}$ . of zein.

used and when mixing was performed at room temperature. The aggregation of particles was not complete immediately on the point of mixing, making the absorbance dependent on the period of time after mixing. A maximum occurred after standing and the time interval was dependent on the concentration of protein. At low concentrations, such as 200 to 400  $\mu\text{g}$ ., the maximum absorbance was reached in about 60 minutes and was practically constant in the 60- to 120-minute range. At 800  $\mu\text{g}$ . the maximum value was reached in 20 minutes and was practically constant in the 20- to 60-minute range.

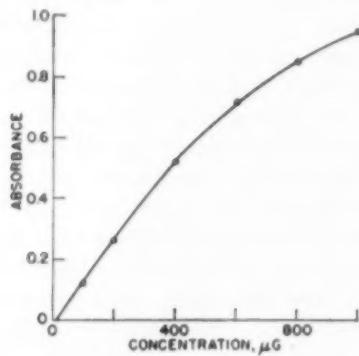


Fig. 2. Absorbance resulting from mixing 2 ml. of 70% ethanol containing varying concentrations of zein ( $\mu\text{g}$ .) with 6 ml. of 1% saline. Time interval: 60 minutes.

Figure 2 shows the relation between absorbance and concentration in a range from 100 to 1000  $\mu\text{g}$ . after 60 minutes. Linearity is confined to the range of about 100 to 500  $\mu\text{g}$ , while beyond 500  $\mu\text{g}$ , the absorbance is not a linear function of concentration. Amiot and Blondeau (1) have attributed this deviation from linearity to secondary reflection from the suspended particles. The results obtained with zein at high concentration gave a constant value of  $k$  when fitted to the equation of Amiot and Blondeau,  $d/cx = kc$ , where  $d$  is the absorbance,  $c$  is the concentration in g. per liter, and  $x$  is the cell thickness in cm.

Although the absorbance was not a linear function of concentration above 500  $\mu\text{g}$ ., the values were reproducible so that with numerous aliquots a standard curve was constructed which did not require daily standardization. Reproducibility at concentrations above 500  $\mu\text{g}$ . was not obtained until it was observed that violent shaking caused the suspended particles to aggregate into larger stringy particles. The absorbance of a suspension containing 1000  $\mu\text{g}$ . dropped from 0.95 to 0.45 by vigorous shaking for 30–40 seconds. When shaking procedures were eliminated, values over the entire range from 100 to 1000  $\mu\text{g}$ . became reproducible. "Blowing in" the precipitant gave sufficient mixing and was not too violent.

From 16 replications at each concentration level of 100, 200, 400, 600, and 800  $\mu\text{g}$ . of zein the turbidities gave average absorbances of 0.10 (standard error<sup>2</sup> 0.006), 0.25 (0.011), 0.50 (0.002), 0.70 (0.005), 0.84 (0.004).

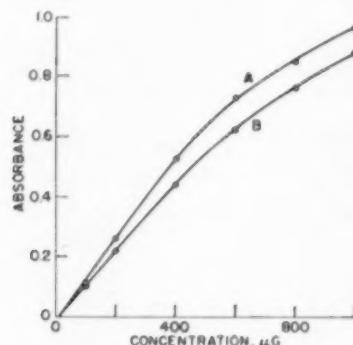


Fig. 3. Effect of ethanol concentration on the particle size resulting from precipitation of zein. Curve A: the 2 ml. of zein were dissolved in 60% ethanol. Curve B: the 2 ml. of zein were dissolved in 90% ethanol. The precipitating agent was 4 ml. of 1% saline. Time interval: 60 minutes.

<sup>2</sup> The standard errors given in parentheses apply to single determinations rather than to the mean absorbances which accompany them.

In most of the experiments where the assay has been used in this laboratory the concentration of ethanol has been constant. The particle size of the zein is dependent on the concentration of ethanol from which it is precipitated when a constant volume of precipitant is used. Figure 3 shows that the absorbance of the zein precipitated from 60% ethanol (A) was higher than that precipitated from 90% ethanol (B) at all concentrations of protein. Where the concentration of ethanol was known, no difficulties occurred; standard curves at various ethanol concentrations have been prepared and are in use in this laboratory. Reproducible results were obtained in a concentration range from 60 to 90% ethanol.

*Nephelometric Assay.* Figure 4 shows the effect of time on the scatter of light at three concentrations of zein. The conditions are not the

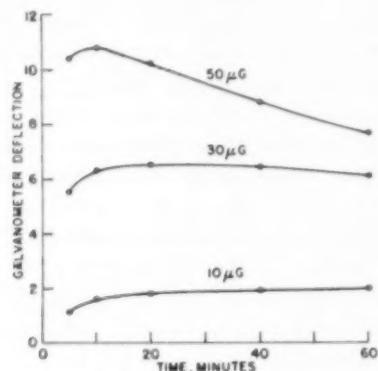


Fig. 4. Relation between light scatter and time interval after mixing. Galvanometer deflection due to scatter of light from zein precipitations was observed after mixing of 2 ml. of zein in 70% ethanol with 4 ml. of 0.1% saline.

same as those described in the suggested procedure but are typical of results with zein. At low concentrations (10  $\mu\text{g}.$ ) aggregation was not complete until 20 minutes after mixing. At higher concentrations (50  $\mu\text{g}.$ ) aggregation was complete in 10 minutes. The rapid decrease in light scatter at the 50  $\mu\text{g}.$  level indicated that a rapid decrease in particle size occurred after precipitation was complete. Figure 5 shows the linear relationship obtained at 10 minutes in a low range (0–40  $\mu\text{g}.$ ) with the volumes chosen for the best results. From 12 to 18 replications at each level of 10, 20, 30, 40, and 50  $\mu\text{g}.$  of zein the scatter of light produced an average galvanometer deflection of 2.1 (standard error<sup>3</sup> 0.14), 4.7 (0.18), 7.1 (0.11), 9.2 (0.18), and 11.2 (0.18).

<sup>3</sup> See footnote 2.

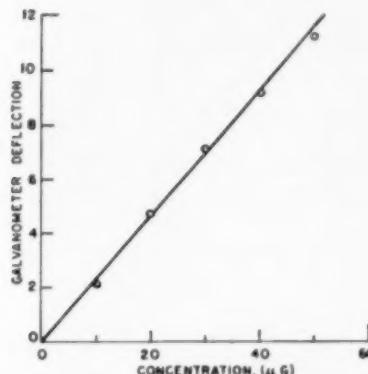


Fig. 5. Relation between concentration and light scatter after 10 minutes. Galvanometer deflection due to scatter of light from zein precipitations was observed after mixing of 1 ml. of zein in 70% ethanol with 6 ml. of 1% saline.

*Dialysis of Zein.* Eight samples of zein, each containing 28.9 mg. of nitrogen, were dialyzed for 24 hours against varying concentrations of alcohol (50, 70, and 95%). The average loss of nitrogen was 0.14 mg. or 0.5% and there was no difference among the three concentrations of alcohol.

### Discussion

When the alcohol concentration of the zein solutions is unknown, prior dialysis against alcohol solutions of known concentrations permits more accurate application of the assay. Two reports in the literature (4, 5) indicate that some zein molecules are sufficiently small to penetrate dialysis membranes. Our tests with commercial zein and several purified laboratory preparations did not substantiate this contention. It appears that in one of the reports (4) the dialysis of amino acids and peptides may account for the observation, but such is not the case in the other (5).

A very definite advantage of this assay procedure is the easy recovery of the material being assayed. This recovery is of particular importance in chromatographic and electrophoresis work. Dialysis against proper ethanol-water mixtures will redissolve the zein and remove the salt impurity.

At present, it is necessary to advocate cautious use of this assay. Some difficulty was experienced with crude ethanol-water extracts of corn which contained zein. In some cases reproducible results were not obtained when the crude extracts were assayed at several concentration levels. Such deviation in the relation between absorbance and

concentration was not obtained in all cases, and it never occurred with dialyzed preparations. It is probable that these difficulties are due to variations in the concentration of ethanol.

Several reports (2, 5, 6) have established the heterogeneity of the fraction of corn protein referred to as zein. Adequate separation of zein into definite and characterizable fractions has not been reported. If there is considerable variation in molecular weight of zein molecules, it is possible that the size of particle produced on introduction of a precipitant will vary. Thus, not all fractions would give the same absorbance per weight of protein. Our present evidence shows no lack of reproducibility with fractions and various preparations; however, considerable work will be necessary to establish this point conclusively. For the present the method is adequate for rapid assay of numerous samples containing zein as is essential in chromatographic and zone electrophoresis experiments. It seems reasonable to assume a similar assay could be used with other prolamines.

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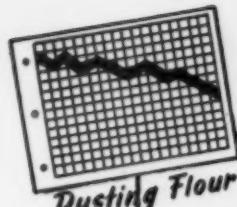


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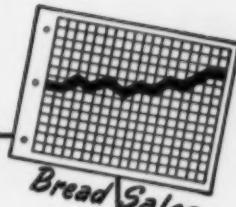
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